TKP4105/TKP4110

Oldershow perforated plate distillation Report

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Conducted on: 15th of October 2012

Summary

A mixture of 11 mol% ethanol in water was distilled in an Oldershow perforated plate column. The time for the system to reach steady state for 40 % reboiler power and total reflux was estimated to 15 minutes. The weeping point was observed for a power duty of 10 %, while the flooding point was not achieved, even with the reboiler power set to 100 %. The vapor velocity was found to be linearly increasing with increasing reboiler power, and no clear relationship was found between the column efficiency and vapor velocity.

Contents

1 Introduction

This experiment was performed as a part of the subject TKP4105, *Separasjon-steknikk*, in the autumn 2012, at NTNU. An 11 mol % mixture of ethanol and water was distilled by Oldershow perforated plate distillation. The purpose was to understand and investigate the principles of distillation and the parameters affecting the operation of distillation columns. The time required to reach steady state condition was determined, as well as the efficiency of the column as a function of the vapour velocity and the relationship between the vapor velocity and the reboiler power duty. The weeping condition of the column was also studied.

2 Theory

2.1 Distillation theory

Distillation is a process where a mixture gets physically separated into two or more products with different boiling points. This is done by partial vaporization of a liquid mixture, and/or by partial condensation of a gas mixture. This results in a liquid phase richer in less volatile component, while the gas phase will be richer in more volatile component.

2.2 Weeping point

When the velocity of the vapor through the perforations of a tray is so low that the liquid *weeps* through the holes of a sieve tray, the condition of the system is called *weeping point*. Massive weeping is known as *dumping*, and results in particularly severe phase maldistribution. When weeping point is reached, the contact between the gas and liquid phase will practically disappear.

2.3 Flooding point

Any countercurrent flow separation device is subject to a capacity limitation due to *flooding*. The phenomenon is related to the ability of the two phases to flow in sufficient quantity in opposite directions past one another and is a result of high vapor velocity. The pressure exerted by the vapor from below balances the gravity head of the liquid, and as a result the liquid starts to build up in the column.

2.4 Column efficiency

The total column efficiency in a distillation column is defined as the ratio of total number of ideal equilibrium stages to the number of actual trays in the column:

$$\text{Column Efficiency} = \frac{N_t - 1}{N_a} \times 100\% \qquad (2.1)$$

N_t is the number of theoretical stages which can be found from a McCabe Thiele diagram, while N_a is the number of actual trays. One stage is deducted from the numerator due to the fact that the reboiler is counted as one stage.

If the outgoing liquid and vapor phase establish an equilibrium with each other, the distillation stage is viewed as an ideal one. To reach a high column efficiency its necessary to obtain a thorough contact between the gas and the liquid.

2.5 Reflux

The condensersystem in the distillation apparatus takes the overhead vapor from the column and liquifies a portion of it to return to the tower as *reflux*. For this particular experiment the distillation column will work with a total reflux, although some samples are taken from the column. The influence on the results these will have will be small, and therefore neglected.

2.6 MacCabe-Thiele Method

The McCabe-Thiele Method is a graphical method for determing the number of ideal (theoretical) stages needed for a given separation of a binary mixture. The main assumption made in this method is that there must be equimolar overflow through the tower between the feed inlet and the top tray, and also the feed inlet and the bottom tray. This assumption leads to a straight operating line. It's based on the assumption that at each stage in the column, the liquid and vapor are in equilibrium. This leads to straight lines for each step in the McCabe Thiele diagrams. At every stage, the heat of vaporization of the liquid is equal to the heat of condensation of the vapors.

Another way to show why the operating line is straight, $y = x$, is to start with the equation for the upper operating line:

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{x_d}{R+1} = \frac{1}{1+\frac{1}{R}}x_n + \frac{x_d}{R+1} \qquad (2.2)$$

where y_{n+1}, x_n and x_d are the ethanol mole fractions of vapor at stage $n+1$, liquid at stage n and liquid in the distillate. One can see that as $R \to \infty$, $y_{n+1} \to x_d$. This is the reason for the use of $y = x$ as the upper operating line. The lower operating line is defined as:

$$y_{m+1} = \frac{L_m}{V_{m+1}}x_m - \frac{W x_W}{V_{m+1}} \qquad (2.3)$$

where y_{m+1} and V_{m+1} is the mole fraction and flow respectively, in the vapor phase at stage $m+1$, L_m the liquid flow from stage number m, , W is the flow of bottom product and x_W is the mole fraction of the bottom product. This gives a lower operating line which is $y = x$ as well, because $W = 0$ and the column is operated at total reflux, so $V = L$.

2.7 Gas Chromatography with FIR (far infrared) sensors

Gas Chromatography (GC) is an analytic technique which is commonly used in many research and industrial laboratories. Several different kinds of samples can be analyzed this way, as long as the compounds are sufficiently thermal stable and volatile enough.

The technique requires a mobile and a stationary phase, whereas the mobile phase, or carrier gas, is an inert gas. The stationary phase consist of a packed column where the packing or solid support itself acts as stationary phase, or is coated with the liquid stationary phase. It's also common to use capillary columns and have the stationary phase cover the walls.

Different compounds can be separarted with GC because of the interaction of the compound with the stationary phase. Stronger interaction leads to a longer time

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intervall before the compound releases and goes through the column. This time is called *retention time* and is unique for each solute[3]

3 Method and apparatus

The experiment is described in the document *Felleslab: Distillation Columns, Separation*[1].

3.1 Apparatus

Figure 3.1 shows a picture of the column used for the experiment. The column has 14 trays and a diameter of 0.07 m.
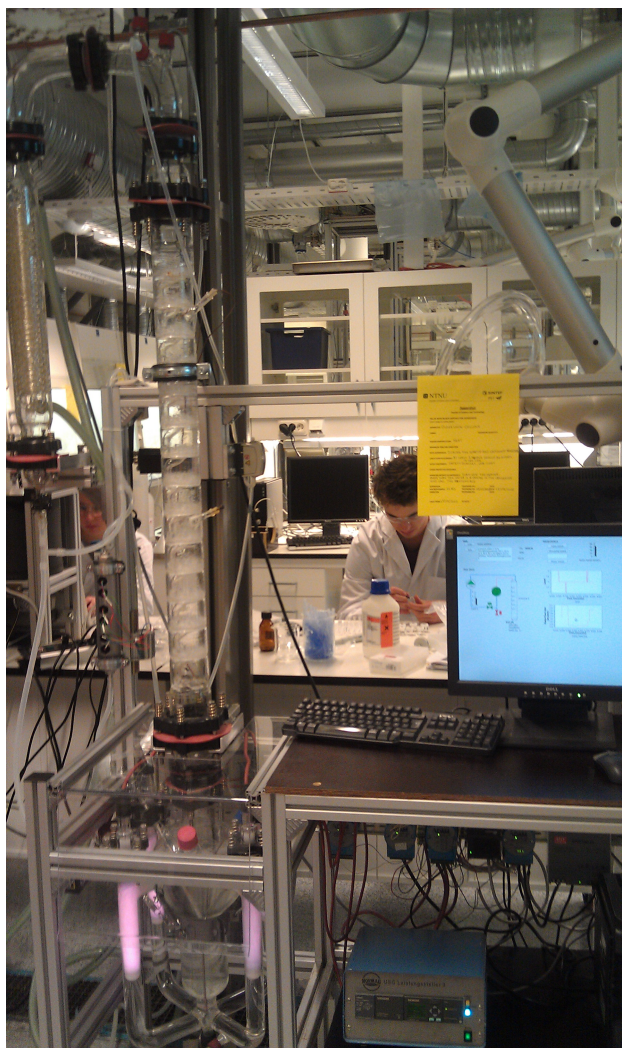

Figure 3.1: A picture of the oldershow distillation column at NTNU.

3.2 Experiment

The experiment was divided into two main parts, the study of approaching steady state and the study of column efficiency vs. vapor velocity. Chemical cards and risk assesment form relevant to the experiment are attached in Appendix D.

3.2.1 The making of ethanol mixture

The column was to be charged with 5800mL of an ethanol-water mixture with $x_{EtOH} = 0.11$. This was made by mixing 1724.17 mL of a solution with 96 vol.% ethanol with 4075.83 mL of water. The calculations that led to these numbers can be found in Appendix B.1. For measuring water, the container used had an accuracy of ± 25 mL. The container used to measure ethanol had an accuracy of ± 10 mL.

3.2.2 Column startup

All the components of the apparatus were turned on, and the column was charged with 5800 mL of the mixture with $x_{ethanol} = 0.11$. The temperature was set to 105°C and the power duty was set to 40%. When the vapor stream started to condense, the time was set to zero by starting the stopwatch.

3.2.3 Time required for reaching steady state condition

When the condensing had started, a top sample was taken out every fifth minute, as shown in appendix E. A bottom sample was also taken after 55 minutes, and the reflux rate at this time was noted. This was used for the next part of the experiment, for a reboiler power duty of 40%. The temperature and pressure at steady state was noted.

3.2.4 Efficiency vs. Vapor velocity

After samples for 40% power duty were obtained, the reboiler power was increased by 10%. Due to the amount of samples that was to be analyzed, and lack of time at the apparatus in the laboratory, the time it took to rach steady state was not found at the day of the experiment. Therefore, an assumed time of 25 minutes

were given by the supervisor. When the steady state was assumed reached, a sample from both top and bottom was taken out and the reflux rate was noted. Then the reboiler power was increased again and the same procedure was followed until measurments for 80% reboiler power was obtained.

3.3 Flooding and weeping point

For the study of flooding point, the reboiler power was set to 100%. Afterwards, the weeping point was studied by setting the reboiler power to 15%, and then decrease it until weeping point was reached.

After these observations, the heater was turned of and the column was shut down.

3.4 Analysis with gas chromatography

In order to analyse the samples, gas chromatography was used. Samples were prepared by pipetting out 250 mL isopropanol and 500 mL of the unknown sample and mix them in a vial. The technique used is described in the document *Felleslab: Distillation Columns, Separation*[1]. The vials were placed in the chromatography apparatus and registered in order to be analyzed.

4 Results

The measured values are shown in Appendix E. Example calculations are shown in Appendix C for column efficiency and vapor velocity, as well as how to determine the number of theoretical steps for a system at steady state.

4.1 Time to reach Steady State

Figure 4.1 shows the volumetric fraction of ethanol in the distillate as a function of time for a reboiler power at 40%. The temperature was 78.3°C, and the pressure in the column was assumed to be equal to the atmospheric pressure, 1 atm. Both the temperature and pressure was assumed constant for all the subsequent

measurements, due to growing errors in the temperature measurements because of liquid inside the insulation of the wires.

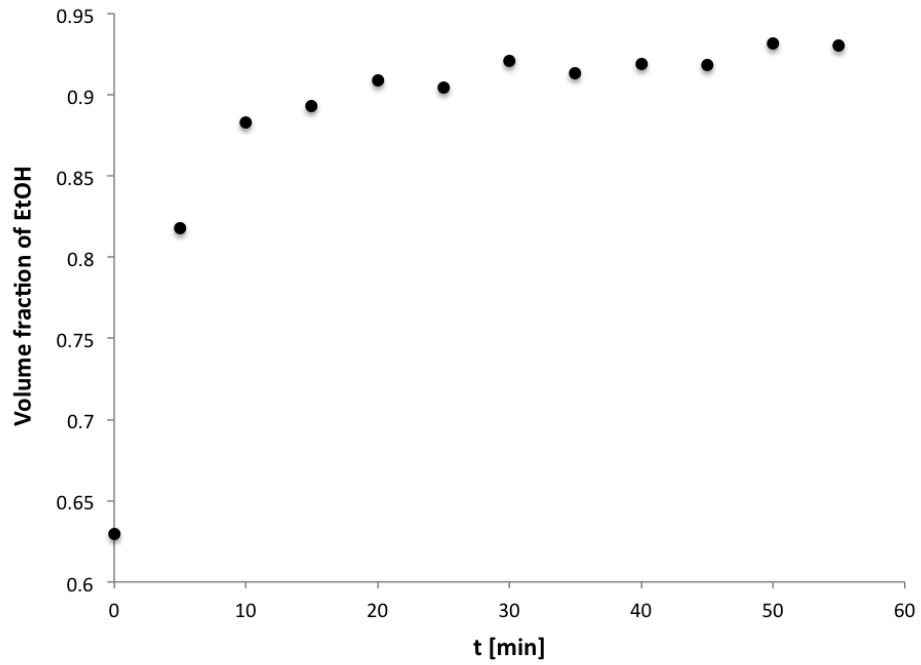Figure 4.1: Volume fraction of ethanol in the distillate as a function of time. The reboiler power was 40%.

4.2 Vapor velocity vs. power duty

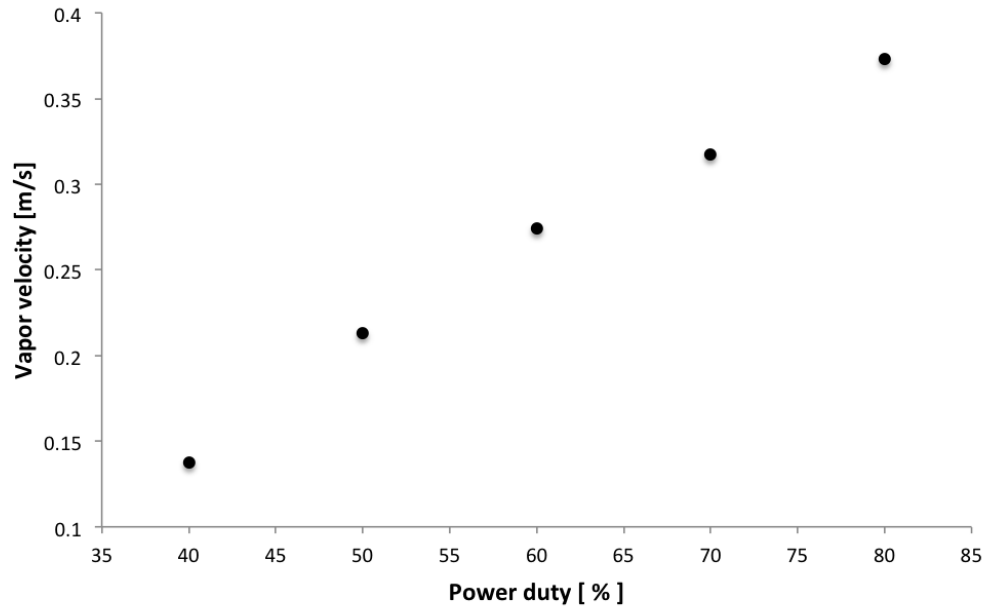Figure 4.2 shows the vapor velocity plotted as a function of reboiler power duty of 40, 50, 60, 70 and 80 percent.

Figure 4.2: Vapor velocity plotted as a function of reboiler power duty for the different steady state measurements at power duites of 40, 50, 60, 70 and 80 percent.

4.3 Vapor velocity vs. column efficiency

Figure 4.3 shows the column efficiency plotted as a function of vapor velocity for 40, 50, 60, 70 and 80 percent power duty. The column efficiency was calculated from Equation (2.1) using a theoretical number of steps aquired from the McCabe Thiele diagrams in Appendix A.
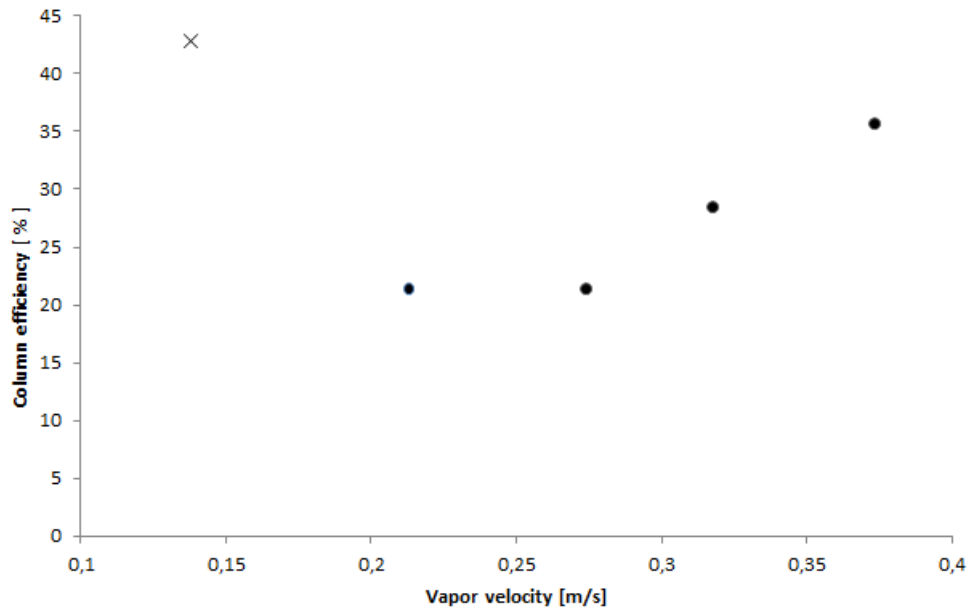
Figure 4.3: Column efficiency at steady state plotted as a function of vapor velocity for power duties of 40, 50, 60, 70 and 80 percent. The first data point is assumed to be a wrong measurement, and therefore marked by x in the diagram.

4.4 Weeping and flooding points

When the reboiler power duty was set to 100 %, the column did not flood. Therefore, no flooding point was observed.

The weeping point was observed at a reboiler power duty of 10 %. After a short while at this power, the top stage drained, and the subsequent stages followed in quick succession.

5 Discussion

The time the system needed to reach steady state for a reboiler power duty of 40 % can be estimated from Figure 4.1. From the graph, the volume fraction of ethanol in the distillate seems to be stabilizing from 15 minutes. There was a slight increase after 15 minutes, however this was relatively small to the slope observed for the first 15 minutes and therefor assumed to be within steady state. This means that the system needs approximately 15 minutes to reach steady state for a reboiler power duty of 40 %.

From Figure 4.2 it is observed that increasing the reboiler power yields a higer gas velocity. From the five measurements that were conducted, the relationship between vapor velocity and reboiler power seems to be directly proporsional. This was expected because an increase in reboiler power is directly proportional to the rate of evaporation, and thereby proportional to the vapor velocity.

The first data point from Figure 4.3 was assumed to be an outlier. Without removing the first data point, it would be hard to draw any conclusion whether the vapor velocity influenses the column efficiency. The trend seems to be that a higher vapor velocity yields a higher column efficiency. However, for even higher vapor velocities than those achieved in the column at max power, the column efficiency would be expected to decrease. In turn, that would give an optimal vapor velocity for the column, achieving a maximun column efficiency.

The flooding point was not observed, even though the power duty was set to maximum. This indicates that the capacity of the column is larger than the gas flow that the reboiler can produce. This means that a more powerful reboiler could have been used, alternatively decreasing the diameter of the column, for educational purposes. The weeping point was observed when the reboiler was set to 10 %. There was a clear shutdown phase for all the trays in the column. This decreases the liquid gas interaction, losing the effect of the trays in the column. After all the trays have shut down, the column would have one equilibrium stage in the reboiler.

5.1 Sources of error

There are a number of sources of error in this experiment. In addition to the expected errors in the appratus with its termometers, pump and sensors, there are a few more worth mentioning. Firstly, the column was assumed to be operating under total reflux. This is not the case, since a a total of 21 samples of high purity ethanol were removed for analysis during the experiment.

The measurements of ethanol content were done using gas chromatography. The GC apparatus in the lab had an uncertainty of about 1 % in its measurements. Along with human inaccuracy with the pipetting, the GC process could amount to a significant source of error. These inaccuracies would effect the number of theoretical stages from the McCabe Thiele graphical method, especially around the top composition because this is close to the azeotrope.

The McCabe Thiele diagrams were made using MATLAB, and the number of theoretical stages was rounded up to the next integer stage. This means that the error in number of theoretical steps from McCabe Thiele could be as much as a whole step. This in turn would influence the column efficiency, and might have contributed to the lacking trend in Figure 4.3.

6 Conclusion

At 40 % reboiler power, it was estimated that the column operated at steady state after approximately 15 minutes. When increasing the reboiler power, it was observed that the gas velocity increased. This relationship was observed to be close to linear. The column efficiency was found to be increasing with increasing vapor velocity. Howerer it was proposed that a maximum vapor velocity would yield an optimal column efficiency, but this was not achieved in the column of interest. Flooding point was not reached, while on the contrary, weeping point was observed at a reboiler power duty of 10 %.

Trondheim, November 2, 2012

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References

[1] Laboratory exercise: *Felleslab: Distillation Columns, Separation, September 2011.*

[2] Chemical card, ethanol: `http: // app. ecoonline. no/ documents/ msds/ 285_ 2991719. pdf`

[3] Theory about Gas Chromatography: `http: // www. chem. ucla. edu/ ~bacher/ General/ 30BL/ gc/ theory. html`

[4] *CRC Handbook of Chemistry and Physics*; 77th ed. CRC Press: Boca Raton, Florida, USA, 1996;:

[5] Gmehling, J. and Onken, *V. Vapour-Liquid Equilibrium Data Collection. Chemistry Data Series*; Vol 1, Part 1, p. 171, Dechema, Frankfort am Main, West Germany, 1977

A McCabe Thiele Diagrams

The following five graphs show the McCabe Thiele diagrams for 40, 50, 60, 70 and 80 % reboiler power, respectively. They were made using MATLAB with equilibrium data from Gmehling and Onken[5], and measurements from Appendix F.

Figure A.1: McCabe-Thiele diagram for the steady state measurment at 40% boiler power duty. Here, $x_{Ethanol}$ is the molefraction of ethanol in liquid phase, while $y_{Ethanol}$ is the mole fraction of ethanol in the gas phase. The bottom composition was $x_{Ethanol} = 0.0895$ and the top composition was $x_{Ethanol} = 0.8057$, and they are marked as circles in the diagram. The equilibrium data is gathered from Gmheling and Onken [5] for a pressure of 1 atm. The operating line is given as $y = x$.
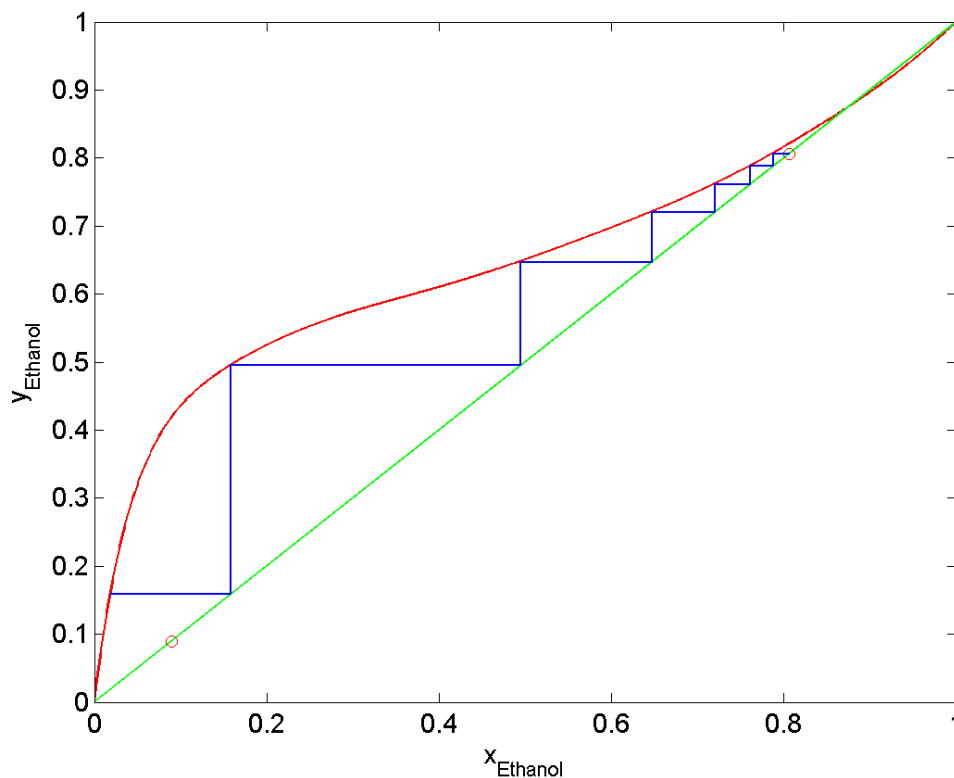
Figure A.2: McCabe-Thiele diagram for the steady state measurment at 50% boiler power duty. Here, $x_{Ethanol}$ is the molefraction of ethanol in liquid phase, while $y_{Ethanol}$ is the mole fraction of ethanol in the gas phase. The bottom composition was $x_{Ethanol} = 0.0849$ and the top composition was $x_{Ethanol} = 0.7315$, and they are marked as circles in the diagram. The equilibrium data is gathered from Gmheling and Onken [5] for a pressure of 1 atm. The operating line is given as $y = x$.

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Figure A.3: McCabe-Thiele diagram for the steady state measurment at 60% boiler power duty. Here, $x_{Ethanol}$ is the molefraction of ethanol in liquid phase, while $y_{Ethanol}$ is the mole fraction of ethanol in the gas phase. The bottom composition was $x_{Ethanol} = 0.0777$ and the top composition was $x_{Ethanol} = 0.7439$, and they are marked as circles in the diagram. The equilibrium data is gathered from Gmheling and Onken [5] for a pressure of 1 atm. The operating line is given as $y = x$.
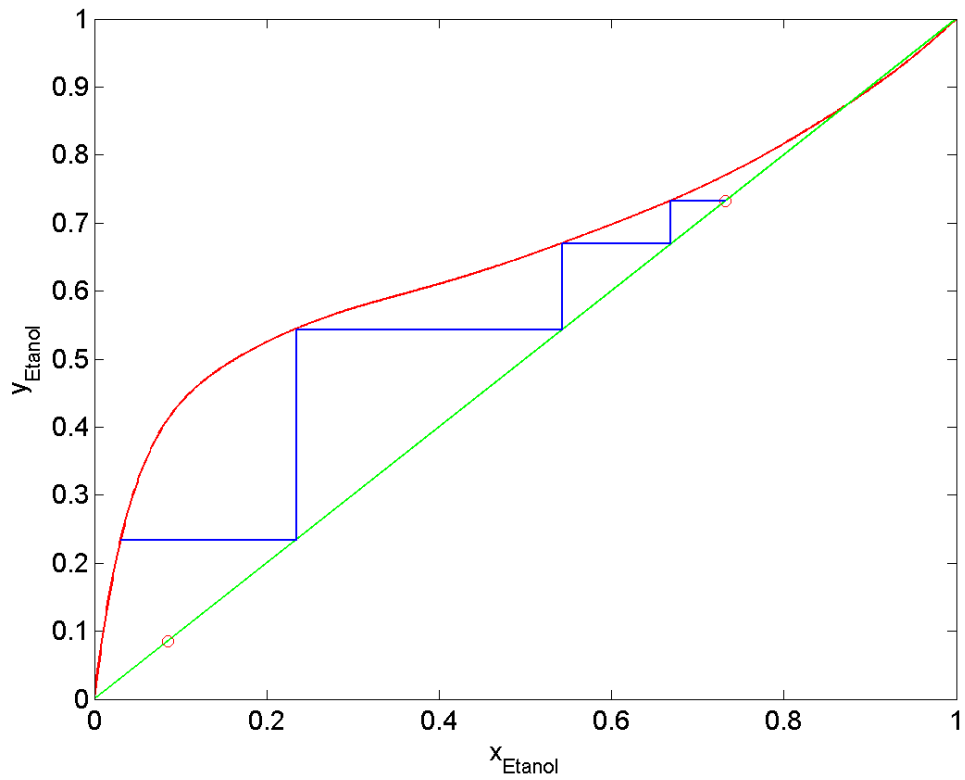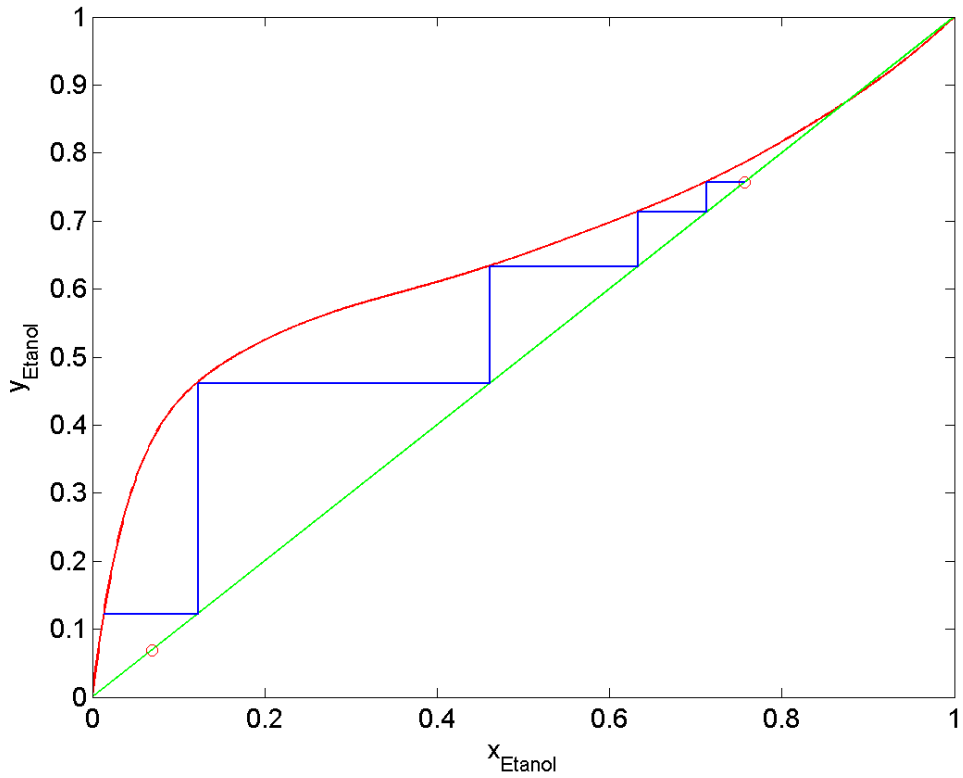
Figure A.4: McCabe-Thiele diagram for the steady state measurment at 70% boiler power duty. Here, $x_{Ethanol}$ is the molefraction of ethanol in liquid phase, while $y_{Ethanol}$ is the mole fraction of ethanol in the gas phase. The bottom composition was $x_{Ethanol} = 0.0692$ and the top composition was $x_{Ethanol} = 0.7564$,and they are marked as circles in the diagram. The equilibrium data is gathered from Gmheling and Onken [5] for a pressure of 1 atm. The operating line is given as $y = x$.
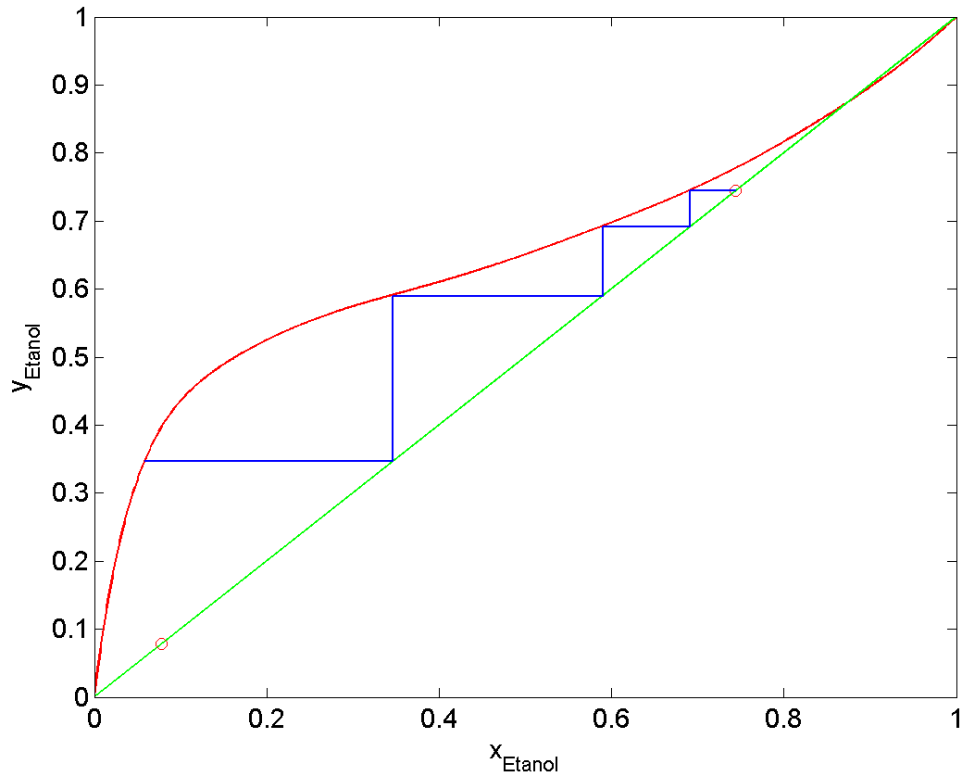
Figure A.5: McCabe-Thiele diagram for the steady state measurment at 80% boiler power duty. Here, $x_{Ethanol}$ is the molefraction of ethanol in liquid phase, while $y_{Ethanol}$ is the mole fraction of ethanol in the gas phase. The bottom composition was $x_{Ethanol} = 0.0626$ and the top composition was $x_{Ethanol} = 0.7892$, and they are marked as circles in the diagram. The equilibrium data is gathered from Gmheling and Onken [5] for a pressure of 1 atm. The operating line is given as $y = x$.
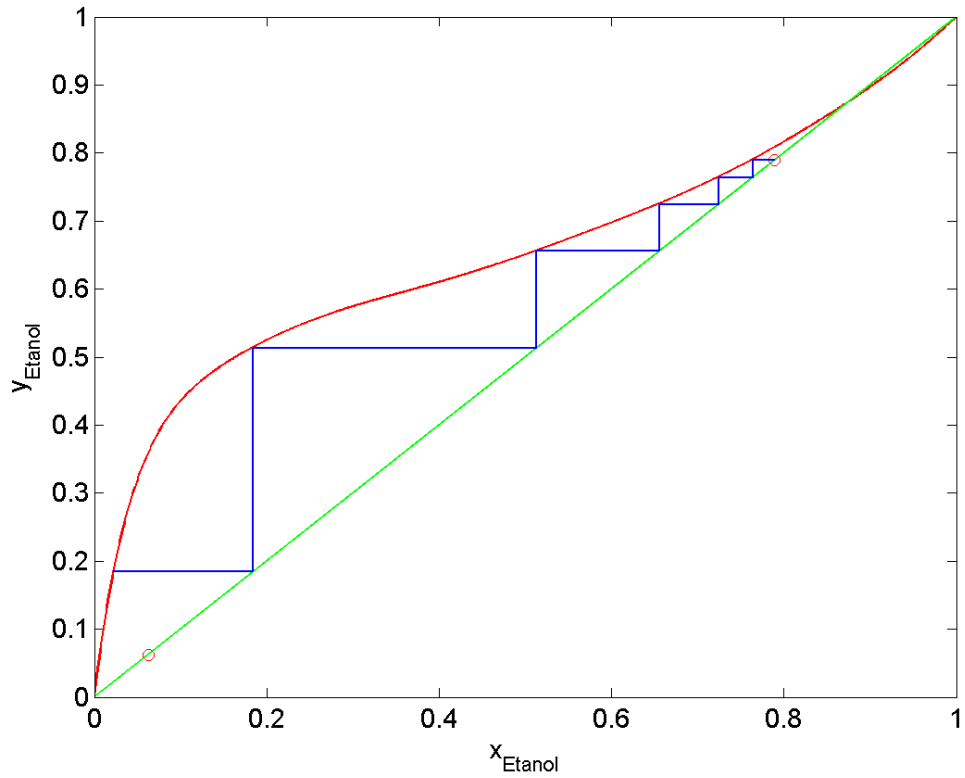
B Calculations

B.1 Calculation required amount of ethanol

The following properties were found using wolfram alpha:

$\delta_{EtOH} = 0.789$ g/cm^3

$M_{EtOH} = 46.0684$ g/mol

$\delta_{H_2O} = 0.997$ g/cm^3

$M_{H_2O} = 18.0153$ g/mol

A solution of 96 vol.% ethanol is handed out, and a solution of 11 mol % ethanol is to be made.

The mole fraction of ethanol in an aqueous solution is given by:

$$x_{EtOH} = \frac{n_{EtOH}}{n_{EtOH} + n_{H_2O}} \tag{B.1}$$

Calculating the number of moles of ethanol is done by:

$$n_{EtOH} = \frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}} \tag{B.2}$$

Analogous for water:

$$n_{H_2O} = \frac{V_{H_2O} \cdot \delta_{H_2O}}{M_{H_2O}} \tag{B.3}$$

Substituting Equation (B.2) and (B.3) into Equation (B.1), gives:

$$x_{EtOH} = \frac{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}}}{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}} + \frac{V_{H_2O} \cdot \delta_{H_2O}}{M_{H_2O}}} \tag{B.4}$$

The volume of water can be calculated using $V_{tot} = V_{EtOH} + V_{H_2O}$. Substituting this into Equation (B.5) gives:

$$x_{EtOH} = \frac{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}}}{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}} + \frac{(V_{tot} - V_{EtOH}) \cdot \delta_{H_2O}}{M_{H_2O}}} \tag{B.5}$$

For this calculation, $x_{EtOH} = 0.11$, and using the data listed over, Equation (B.5) can be solved for V_{EtOH}. This yeilds the volume of pure ethanol needed:

$$V_{EtOH,\ 100\ \%} = 1655.2 \text{ mL} \tag{B.6}$$

Converting the answer to 96 vol % ethanol is done by:

$$V_{EtOH,\ 96\ \%} = \frac{1655.2 \text{ mL}}{0.96} = \underline{\underline{1724.17 \text{ mL}}} \tag{B.7}$$

This means that the required volume of water to make the given solution is:

$$V_{\text{H}_2\text{O}} = V_{tot} - V_{EtOH} = 5800 \text{ mL} - 1724.17 \text{ mL} = \underline{\underline{4075.83 \text{ mL}}} \tag{B.8}$$

B.2   Assignment

These measurments were given in the assignment:

- Weight of empty pycnometer 1: 11.98170 g

- Weight of empty pycnometer 2: 11.77385 g

- Weight of pycnometer 1 with water: 16,95955 g

- Weight of pycnometer 2 with water: 16.75745 g

- Weight of pycnometer 1 with distillate: 16.0903 g

- Weight of pycnometer 2 with bottom product: 16.6962 g

From the GC anaylsis, the following data are found:

- Distilate composition: $x_{EtOH} = 0.744$

- Bottom composition: $x_{EtOH} = 0.033$

First step is to find the volume of each pycnometer, by using the weight of pure water, and the known density of water, $\rho_{water} = 0.997 \text{ cm}^3/\text{mL}$. This gives:

$$V_1 = 4.992\ cm^3 \qquad V_2 = 4.998\ cm^3 \tag{B.9}$$

Then the densities of the distillate and bottom product are found using:

$$\rho = \frac{\text{weight of sample}}{\text{volume pycnometer}} \tag{B.10}$$

This gives the densities:

$$\rho_{\text{distillate}} = 0.8229 \ g/cm^3 \qquad \rho_{\text{bottom product}} = 0.9847 \ g/cm^3 \tag{B.11}$$

From data in *Green and Perry*, the weight % of the distilate is estimated to 88.1 wt.% and of the bottom product 8.0 wt.%. The mole fraction of ethanol in water is:

$$x_{EtOH} = \frac{n_{EtOH}}{n_{EtOH} + n_{H_2O}} \tag{B.12}$$

Using the fact that $n_i = \frac{m_i}{M_i}$, converting the number of moles to mass gives:

$$x_{EtOH} = \frac{\frac{m_{EtOH}}{M_{EtOH}}}{\frac{m_{EtOH}}{M_{EtOH}} + \frac{m_{H_2O}}{M_{H_2O}}} \tag{B.13}$$

Substituting the mass with weight % gives the following equation:

$$x_{EtOH} = \frac{\frac{wt.\%_{EtOH}}{M_{EtOH}}}{\frac{wt.\%_{EtOH}}{M_{EtOH}} + \frac{(100 - wt.\%_{EtOH})}{M_{H_2O}}} \tag{B.14}$$

Substituting for wt. $\%_{EtOH}$ for the bottom product and the distilate gives:

$$x_{EtOH, \text{ bottom product}} = \underline{0.0328}$$

$$x_{EtOH, \text{ distilate}} = \underline{0.7433}$$

Calculating the gas velocity of the vapor in the column is done by using Equation (B.15). The reflux rate is given, 53 mL/min, the temperature is 25°, and the presure is 1 bar:

$$\nu = \frac{V_{gas}}{A \cdot t} \ [\text{m/s}] \tag{B.15}$$

where A is the cross section area of the distillation column, t is the time, and V_{gas} is the volume of the gas which can be found, assuming one can use the ideal gas law:

$$V_{gas} = \frac{n_{gas} \cdot R \cdot T}{P} \tag{B.16}$$

A general mass balance for the two components in the column:

$$m_{tot} = m_{EtOH} + m_{H_2O} = x_{EtOH} \cdot n_{tot} \cdot M_{EtOH} + (1 - x_{EtOH}) \cdot n_{tot} \cdot M_{H_2O} \tag{B.17}$$

Rearranging this for n_{tot} gives:

$$n_{tot} = \frac{m_{tot}}{x_{EtOH} \cdot M_{EtOH} + (1 - x_{EtOH}) \cdot M_{H_2O}} \tag{B.18}$$

Knowing that the reflux ratio is 1, and that $m_{tot} = \rho \cdot V_{tot}$, this can be substituted into Equation (B.19), to give:

$$n_{tot} = \frac{\rho \cdot V_{tot}}{x_{EtOH} \cdot M_{EtOH} + (1 - x_{EtOH}) \cdot M_{H_2O}} \tag{B.19}$$

This will give, using $\rho_1 = 0.8229$ for the distillate, the total number of moles:

$$n_{tot} = 1.1215 \ [\text{mol/min}] \tag{B.20}$$

Substituting this into Equation (B.16) yields:

$$V_{gas} = \frac{1.1215 \ [\frac{mol}{min}] \cdot 8.314 \ [\frac{m^3 Pa}{K \cdot mol}] \cdot (79 + 273) \ [\text{K}]}{100.000 \ [\text{Pa}]} = 0.0328 \ m^3/\text{min} \tag{B.21}$$

Substituting into Equation (B.15), with a column diameter of 0.07 m, gives the gas velocity of the column:

$$\nu = \frac{0.0328 \ [\frac{m^3}{min}]}{\frac{\pi}{4} 0.07^2 \ [m^2] \cdot 60 \ [s]} = \underline{\underline{0.1420 \ [m/s]}} \tag{B.22}$$

C Example calculations

For this example calculation, the numbers and diagrams used will be from the test where the reboiler power duty was set to 80 %. The data used can be found in Appendix E.

The volume percent of the distillate was 92.37 % and for the bottom product it was 17.75 %. Using the following equation, this gave the following mole fractions for ethanol: $x_D = 0.7892$ and $x_B = 0.0626$.

$$x_{EtOH} = \frac{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}}}{\frac{V_{EtOH} \cdot \delta_{EtOH}}{M_{EtOH}} + \frac{V_{H_2O} \cdot \delta_{H_2O}}{M_{H_2O}}} \tag{C.1}$$

In Figure A.5, the top and bottom compositions are shown as small red circles. Then the McCabe Thiele graphical method is begun, making steps between the equlibrium line, and the operating line. From Figure A.5 a number of 5.7 theoretical stages is estimated. Since stages are integers, this is rounded up to 6 by MATLAB.

Using the equation given below, with the number of theoretical tray $N_t = 6$ and $N_a = 14$. This gives a column efficiency of 35.7 %.

$$\text{Column Efficiency} = \frac{N_t - 1}{N_a} \times 100\% \tag{C.2}$$

For calculating the vapor velocity, the volume of gas travelling up the column must be calculated. This is done by converting the reflux rate of the distillate to the equivalent volumetric flow rate in gas phase. When the total gas phase volumetric flow is known, this is then divided by the cross section area of the column to give the vapor velocity. Following this procedure yields a vapor velocity of 0.373 m/s.

D Risk assessment and chemical data sheets

Data necessary to fill out the chemical data sheets was found from *ECOonline* [2]

SINTEF   **PFI**   **NTNU**
Norges teknisk-
naturvitenskapelige
universitet

Chemical card

	NAME Ethanol					FORMULA C_2H_5OH	
							HEALTH HAZARDS No special health risk.
PHYSICAL DATA	Melt.p./ fr.p.	Boil.p	Fl. p.	Molar mass	Expl.lim.		FIRE HAZARDS Extremely inflammable
	-114 °C	78 °C	363 °C	46,07 g/mol	3,3-19,0%		REACTION HAZARDS Avoid alkali metals, ammonia, oxidizing agents and peroxides.
PROTECTIVE MEASURES	Use respirator if necessary. Use safety gloves. Use safety goggles. Choose other safety equipment that suits the risk of your experiment.					ADMINISTRATIVE NORM	
HELSEFARE Inhaling	May be harmful. May lead to irritation of the air passage.					FIREFIGHTING "Alcohol"-foam, dry chemicals or carbon dioxide. Water as fog or spray.	
Ingestion	May be harmful.						
Skin	May be harmful if absorbed through the skin. May cause skin irritation.						
Eyes	May cause eye irritation.						

First aid	EYES Rinse with water. SKIN Wash with soap and water. Contact doctor.	INGESTION Do NOT induce vomiting. Rinse mouth with water. Contact doctor. INHALING Move person to fresh air. Provide artificial breath if needed. Contact doctor.

SPECIAL MARKS	
WASTE	Contact a licensed treatment- or terminal station for hazardous waste for destruction of this material. Use a incinerator.
STORAGE	Keep away from fire sources. Keep in a cool, dry and ventilated place.

Kjemikaliekort

	Name: *Distilled water*			Formula: H_2O	
					Health risk: -
Physical data	Molar mass	Melt.p./ fr.p.	Boiling point	Fire risk: -	
	18.0153 g/mol	0 ℃	100 ℃		

NTNU		Risikovurdering	Nummer	Dato
HMS		HMS-avd.	HMSRV2601	
		Godkjent av	Side	Erstatter

Hazardous activity identification process

Unit: _Kjemisk prosessteknologi_   **Date:**   15.10.2012

Line manager: _Øyvind Gregersen_

Participants in the identification process (including their function): _Elise Landsem and Audun Formo Buene (Group B19)_

Short description of the main activity/main process:

ID no.	Activity/process	Responsible person	Laws, regulations etc.	Existing documentation	Existing safety measures	Comment
1	Distillation of ethanol	V.L.M.		Felles Lab: Distillation Columns	Safety goggles, lab coat	
2						
3						

NTNU		Risk assessment		Utarbeidet av	Nummer	Dato
				HMS-avd.	HMSRV2603	04.02.2011
HMS /KS				Godkjent av	Side	Erstatter

Unit: *Kjemisk prosessteknologi*   **Date:** 10.10.2012

Line manager: *Øyvind Gregersen*

Participants in the identification process (including their function): *Elise Landsem and Audun Formo Buene (Group B19)*

Signatures:

ID no.	Activity from the identification process form	Potential undesirable incident/strain	Likelihood: Likelihood (1-5)	Consequence: Human (A-E)	Consequence: Environment (A-E)	Consequence: Economy/ material (A-E)	Consequence: Reputation (A-E)	value Human	Comments/status Suggested measures
1	Distillation of ethanol	Fire	2	B	A	A	B	2B	Avoid open flames. Have fire extingushing equipment ready.
2	Distillation of ethanol	Explosion	1	B	A	A	B	1B	Avoid open flames. Have fire extingushing equipment ready.
3	Distillation of ethanol	Poison	1	A	A	A	A	1A	Avoid inhalation of fumes. Ensure proper ventilation.
4	Gas chromat	Gass leak	2	C	A	B	B	2C	Good maintainance of equipment. Check for leaks.

E   Lab journal

LABJOURNAL: Time required for reaching steady state condition, measurments

vol% EtOH

Sample nr.	t [min]	X̶E̶t̶h̶a̶n̶o̶l̶	X̶w̶a̶t̶e̶r̶	Comments
1	5̶ 0	0,6298		taken after some minutes (3), but doesn't matter due to acumulation.
2	1̶0̶ 5	0,8181		Actually, after 6 min.
3	1̶5̶ 10	0,8831		OK
4	2̶0̶ 15	0,8934		OK
5	2̶5̶ 20	0,9091		OK
6	3̶0̶ 25	0,9045		OK
7	3̶5̶ 30	0,9212		OK
8	4̶0̶ 35	0,9136		OK
9	4̶5̶ 40	0,9189		OK
10	5̶0̶ 45	0,9186		OK
11	5̶5̶ 50	0,9318		OK
12	6̶0̶ 55	0,9306		OK
13 (Bottom)	6̶0̶ 55	0,2412		OK

Estimated time until steady state were reached: _____[min]

Other comments:

Measurement volume: ± 10mL for ethanol
                                    ± 25mL for water

Leackage during the heatup! new sample!
May be source of error: Some rests from 1. attent still in the reboiler when filling up again!
Number of trays: 14

LABJOURNAL: Efficiency vs. Vapor velocity

Measurment number	Boiler power	Reflux rate mL/min	$X_{Ethanol}$ frac. (vol%)	$y_{Ethanol}$ frac. (vol%)	Comments
1	40%	55	0,2412	0,9306	Same numbers as in steady-state measure
2	50%	80	0,2307	0,8981	flow rate 80 ml/min
3	60%	104	0,2142	0,9038	
4	70%	121,8	0,1937	0,9094	
5	80%	147,0	0,1775	0,9237	

For calculations:
$D_{column}=$ __7__ [cm]= _0,07_ [m]

Other comments:

$T_{steady\ state} = 78,3°C$
$P_{steady\ state} = 1atm$

14: top 50%
15: btm 50%
16: top 60%
17: btm 60%
18: top 70%
19: btm 70%
20: top 80%
21: btm 80%

Weeping point:

Pressure drop	$\frac{o}{?}$
Vapor velocity	$\frac{o}{.}$
Boiler power	10%

Other comments:

Weeping point was observed.
at 100% - flooding point not observed, to high
capacity!

F   Excel calculations

Figure F.1 shows the ecxel page for calculating the vapor velocity and the column efficiency for the five different reboiler power duties.

Reflux [mL/min]	vol% EtOH frac	vol EtOH (l) mL/min	vekt EtOH g/min	Vekt vann g/min
55	0,9306	51,183	40,3987419	3,8101294
80	0,8981	71,848	56,7096264	8,1373264
104	0,9038	93,9952	74,1904114	9,98679136
121,8	0,9094	110,76492	87,4267514	11,0152169
147	0,9237	135,7839	107,174232	11,195911

mol EtOh mol/min	mol vann mol/min	mol tot mol/min	Vgas m^3/min	Vgas m/sek
0,87692956	0,21149409	1,08842365	0,03180325	0,13773183
1,23098754	0,45168975	1,68267729	0,04916708	0,21293016
1,61044037	0,55435054	2,16479092	0,06325422	0,27393802
1,89775967	0,61143677	2,50919644	0,0733176	0,31751995
2,32641534	0,62146681	2,94788215	0,0861358	0,37303233

density EtOH g/mL	density H2O g/mL	Mm EtOH g/mol	Mm H2O g/mol
0,7893	0,9982	46,0684	18,0153
R [m^3atm/K]	Areal	T [K]	P [atm]
8,314	0,00384845	351,45	1

Vgas m/sek	Column eff.	Nt
0,13773183	42,8571429	7
0,21293016	21,4285714	4
0,27393802	21,4285714	4
0,31751995	28,5714286	5
0,37303233	35,7142857	6

Figure F.1: Excel page showing the numbers from the calculations of vapor velocity and column efficiency.