FROM LAB TO DIGITAL TWIN

ESTIMATING SIMULATION PARAMETERS FOR BATCH COLUMNS FROM EXPERIMENTAL DATA

For more than 30 years, chemical engineers have relied on CHEMCAD, a leading chemical process simulation software, to solve industry challenges. This paper outlines how CHEMCAD is helping engineers make advances in batch distillation.

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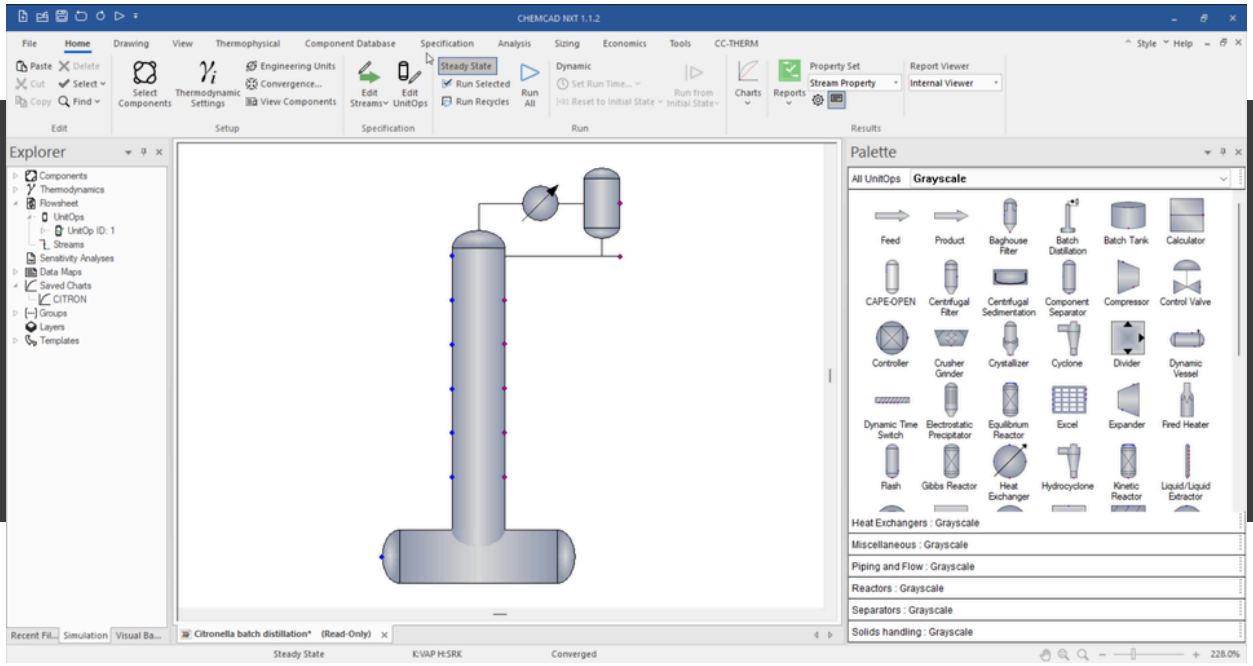
Estimating Simulation Parameters for Batch Columns from Experimental Data

Jan Schöneberger



Introduction

The Batch Column in CHEMCAD is a powerful unit operation that allows the fast, simple, and reliable estimation of the feasibility of a batch distillation process. Whether you're designing a new column, developing new separation processes in existing columns, optimizing current plants, or troubleshooting issues, this unit operation can perform all these functions and more. The objective of this paper is to introduce and find the parameters required for creating the Digital Twin of an existing batch distillation plant. The considered plant is a batch column allocated in the laboratory for Thermal Process Technology of the Berlin University of Applied Science (BHT). However, the presented methodology can be applied to a plant of any size.

The Separation Problem

Three liters of an ethanol water mixture were produced through a fermentation process. The yeast was already separated, and the composition was expected to be around eight weight percent of ethanol. Methanol and residual substrates are considered negligible. The ethanol concentration should be increased as much as possible to save energy in the subsequent separation step, where almost pure ethanol is withdrawn in an adsorption process.

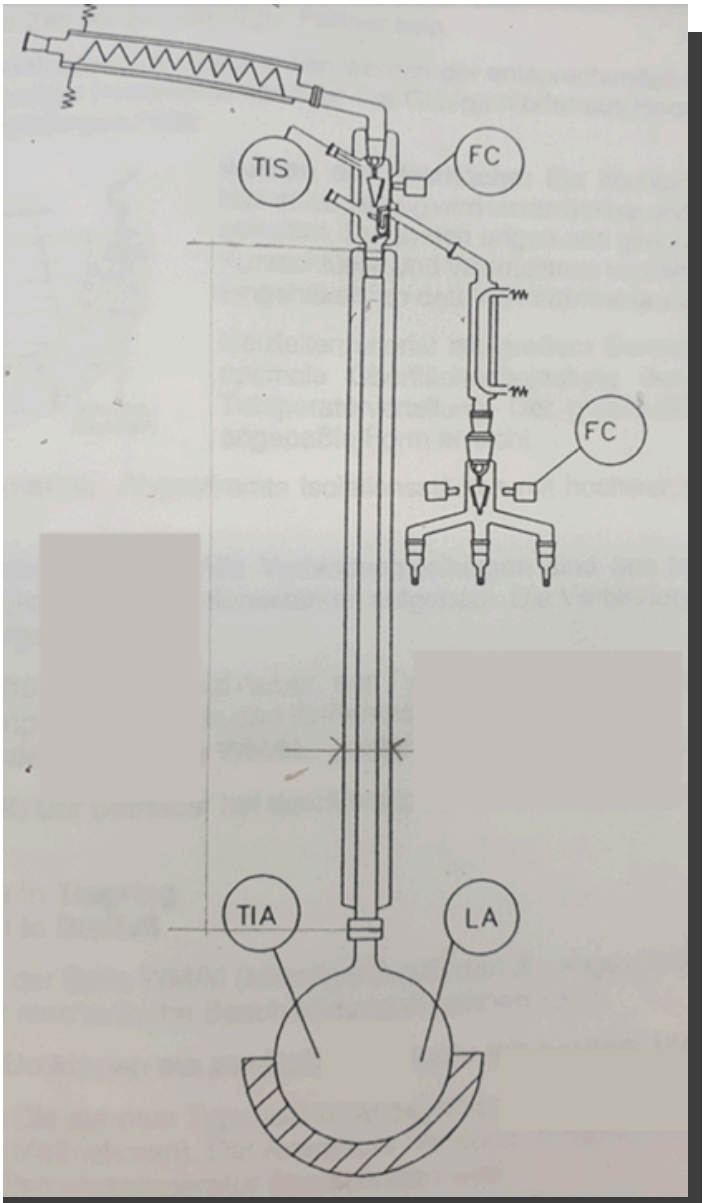


Figure 1: Sketch of the batch distillation plant.

Description of the Plant

The plant that is used to solve this separation problem is composed of a pot with a volume of four liters, a column with an internal diameter of 50 mm and a height of 1200 mm, a condenser with integrated reflux control by a magnetic valve, a guard condenser, a product cooler, and three receiver tanks, that can be connected by a magnetic valve. The receiver tanks can be replaced whenever necessary.

The column is filled with Raschig rings. All parts are made of glass. A sketch of the plant is depicted in Figure 1. The pot, the column, and the condenser are isolated against heat losses. A photo of the plant can be found in Figure 2.

The pot is heated with a controllable heat mantel which has a maximum electricity uptake of 1800 Watt. However, with heat duties above 600 Watt, flooding in the condenser was observed for ethanol water mixtures around eight weight percent.

Temperature measurements are available in the pot, in the middle of the column, at top of the column, and in the condenser. The cooling water flow rate is measured together with inlet and outlet temperatures. The reflux is controlled via the opening time of a magnetic flow split valve.

All signals are captured electronically and send to a Lab View based distributed control system. A screenshot of the graphical user interface is given in Figure 3.

Figure 2: Batch distillation plant in the TPT lab of the Berlin University of Applied Science.

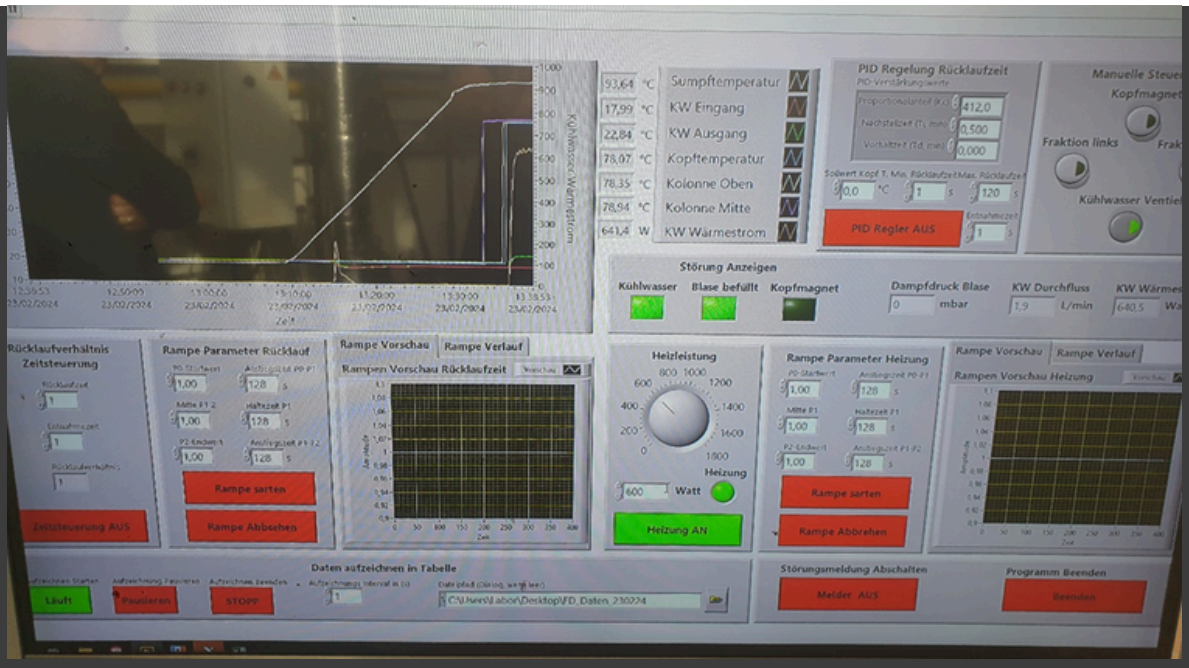


Figure 3: Screenshot of the distributed control system in Lab View.

The geometric and technical parameters that are relevant for the simulation of the plant are summarized in Table 1.

Table 1: Geometric and technical parameters of the batch distillation plant.

Column		Pot	
Height	1200 mm	Heat duty (max)	1800 W
Inner diameter	50 mm	Heat duty (flood)	600 W
Packing (Raschig Rings)	**1000 mm**	Volume (max)	4 L
Length	15 mm	Volume (used)	3 L
Diameter	15 mm	**Receiver Tanks**	**3**
Thickness	2 mm	Volume (max)	0.25 L

First Feasibility Check

A rough estimation of the HETP value for the packing of 500 mm is used for the first feasibility check. This gives two theoretical plates plus the condenser and the pot, and we can set-up the first simulation in the following way:

1. Adjust engineering units. (min kg °C bar kW-h mm l)
2. Add ethanol and water to the component list.
3. Accept the model selections (NRTL + Latent Heat).

Figure 4 : First Feasibility Check - Steps 1 to 3.

4. Move the batch column from the palette to the flowsheet.
5. Double click on the column to start the specification. Remark: When you specify the UnitOp for the first time, the main menu does not appear and CHEMCAD leads you automatically through the screens.
6. Specify the pot charge. Press “Flash” to get the boiling point temperature.

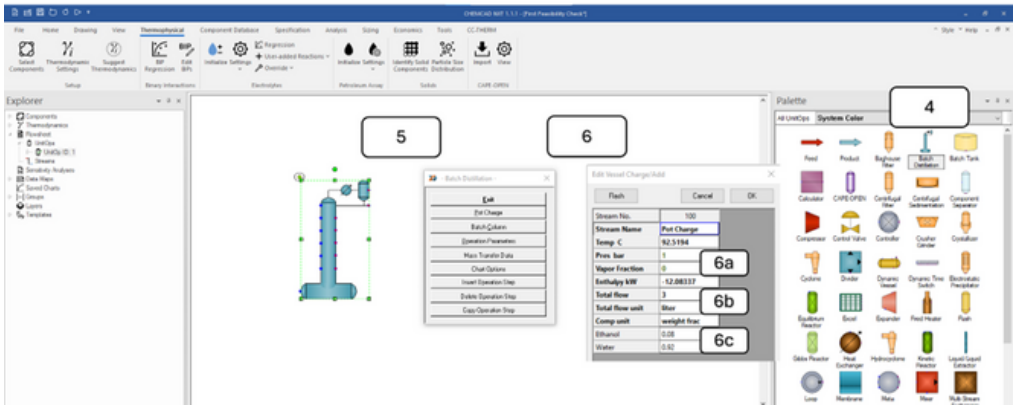


Figure 5 : First Feasibility Check - Steps 4 to 6.

7. Specify the batch column. (Number of stages 4, Number of operation steps 1)

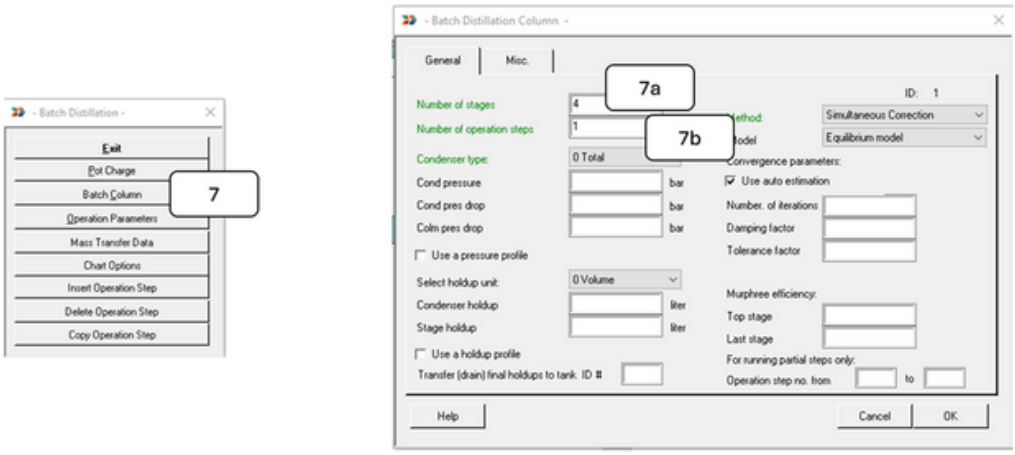


Figure 6 : First Feasibility Check - Step 7

8. Specify the first operation step. (Reflux ratio 10, Reboiler duty 1 kW)

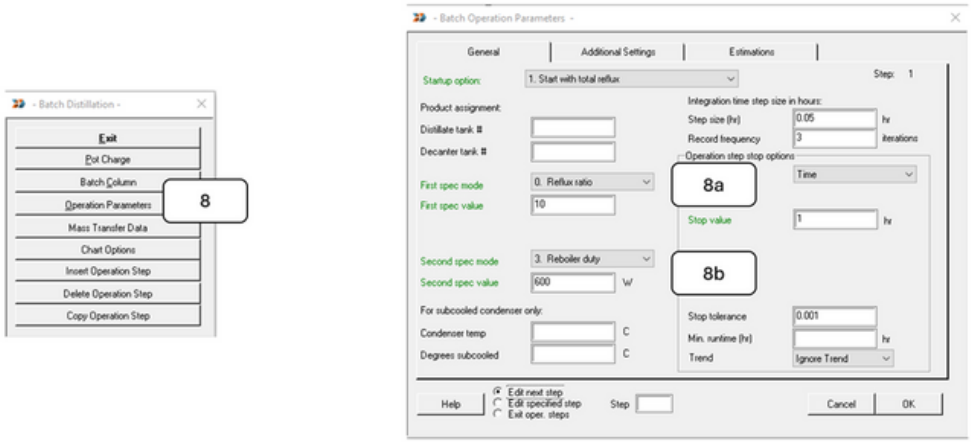


Figure 7 : First Feasibility Check - Step 8

9. Select the chart options.

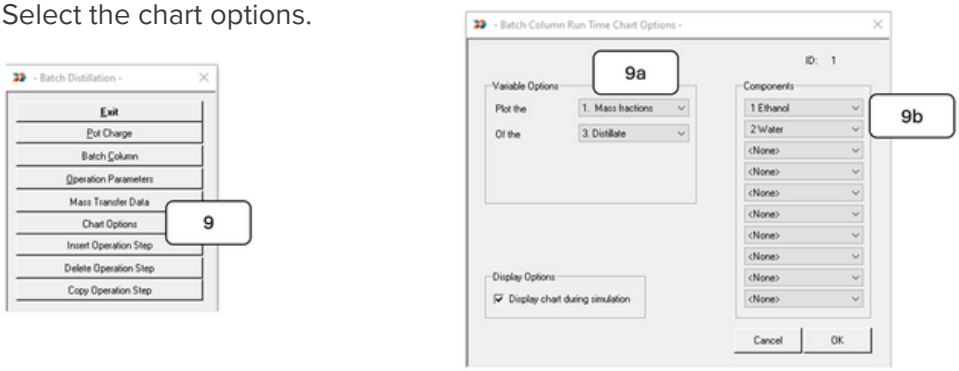


Figure 8 : First Feasibility Check - Step 9

10. Run the flowsheet.

CHEMCAD plots the selected variables in a live chart, see Figure 9.

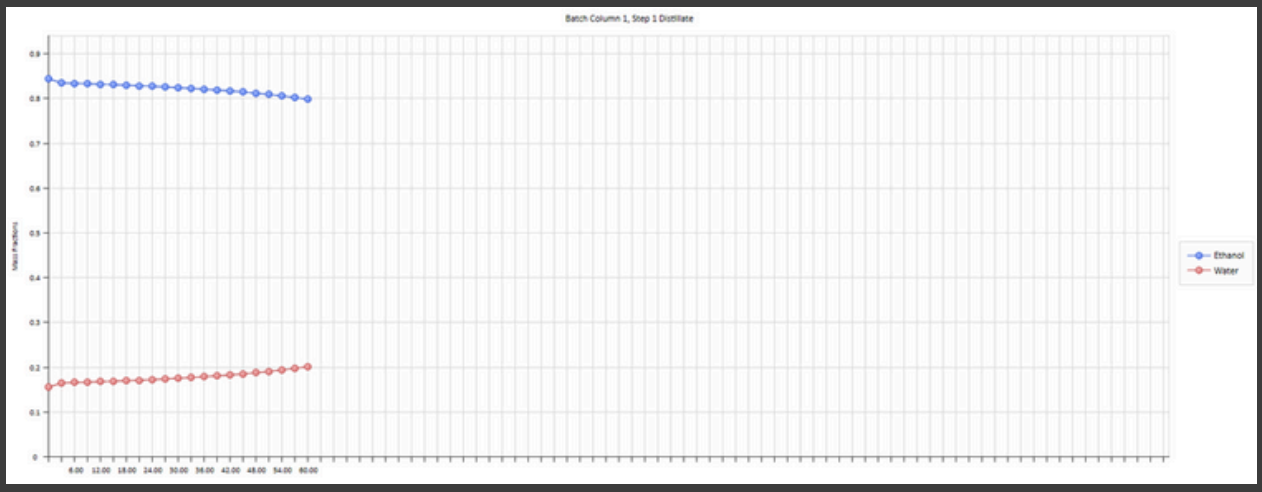


Figure 9 : Live chart of the distillate concentration based on the parameters set for the first feasibility study.

Let's analyze these first results!

To get some more detailed plots, go to Charts and select Batch Column (Figure 10).

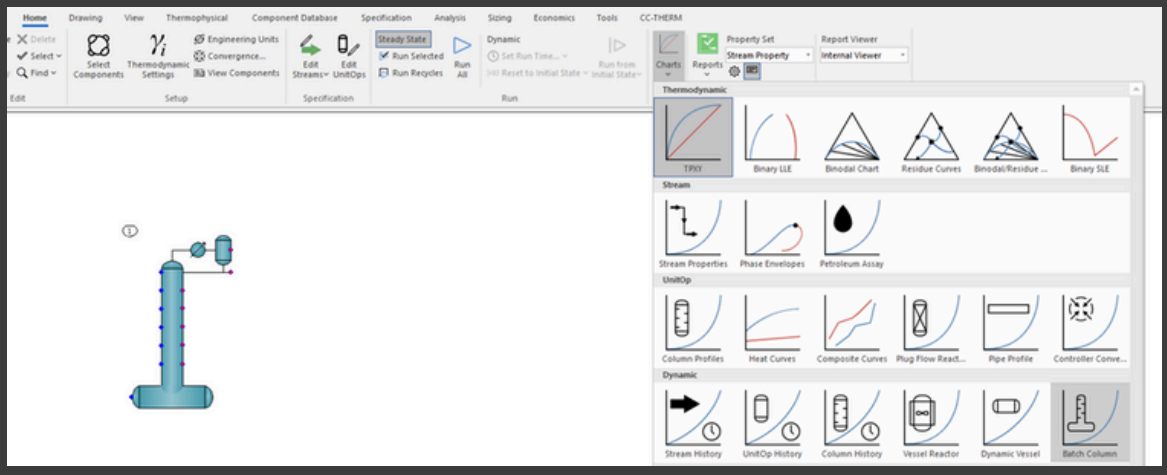


Figure 10 : Access to the graphical results of the batch column distillation simulation.

Plot the ethanol mass fraction in the distillate and repeat the procedure to plot the ethanol mass in the bottom. Figure 11 shows the results when both plots are tiled horizontally.

The first bit of information that we can get from this is the maximum ethanol concentration achieved in the distillate is slightly above 84 weight percent. The azeotropic point of an ethanol water mixture of one bar is at approximately 96 weight percent.

After 50 minutes the distillate concentration starts to drop faster and reaches values below 80 weight percent. At this time point, around 54 % of the ethanol has been recovered.

Here, the reflux ratio was maintained at a constant value of 10. An alternative strategy is to increase the reflux ratio in order to maintain the distillate concentration.

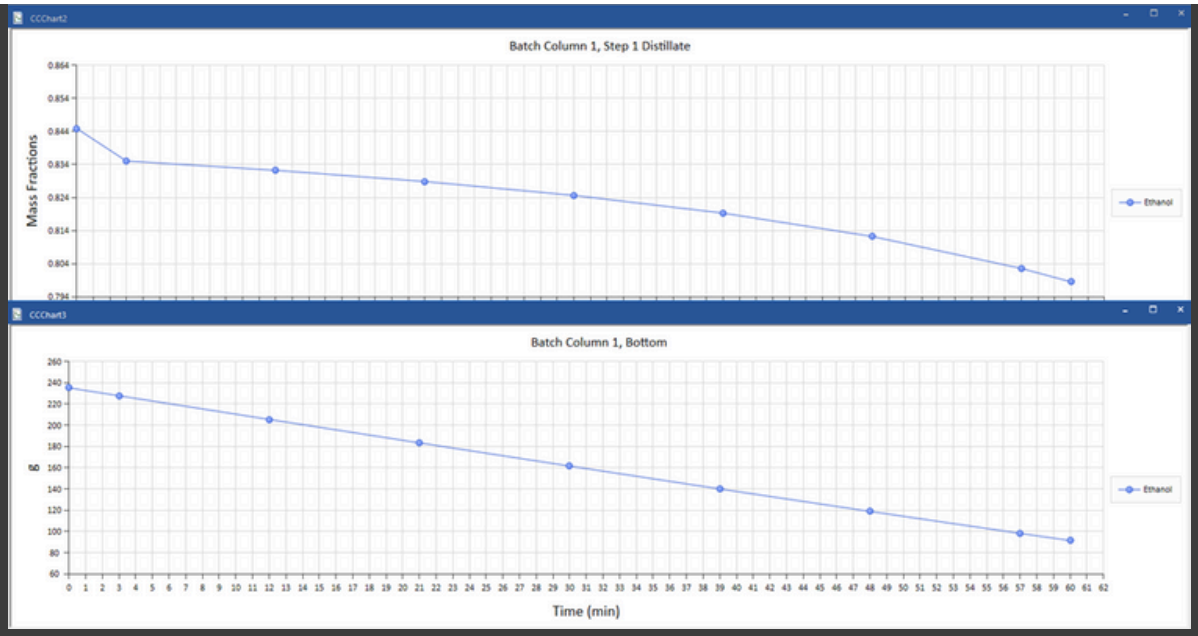


Figure 11 : Remaining ethanol mass in the pot and ethanol mass fraction in the distillate.

To evaluate this scenario, the operation step is changed according to Figure 12. CHEMCAD now adjusts the reflux ratio to get a constant 84 weight percent (step 11a). The calculation should stop when the distillate flow drops below one gram per minute (step 11b).

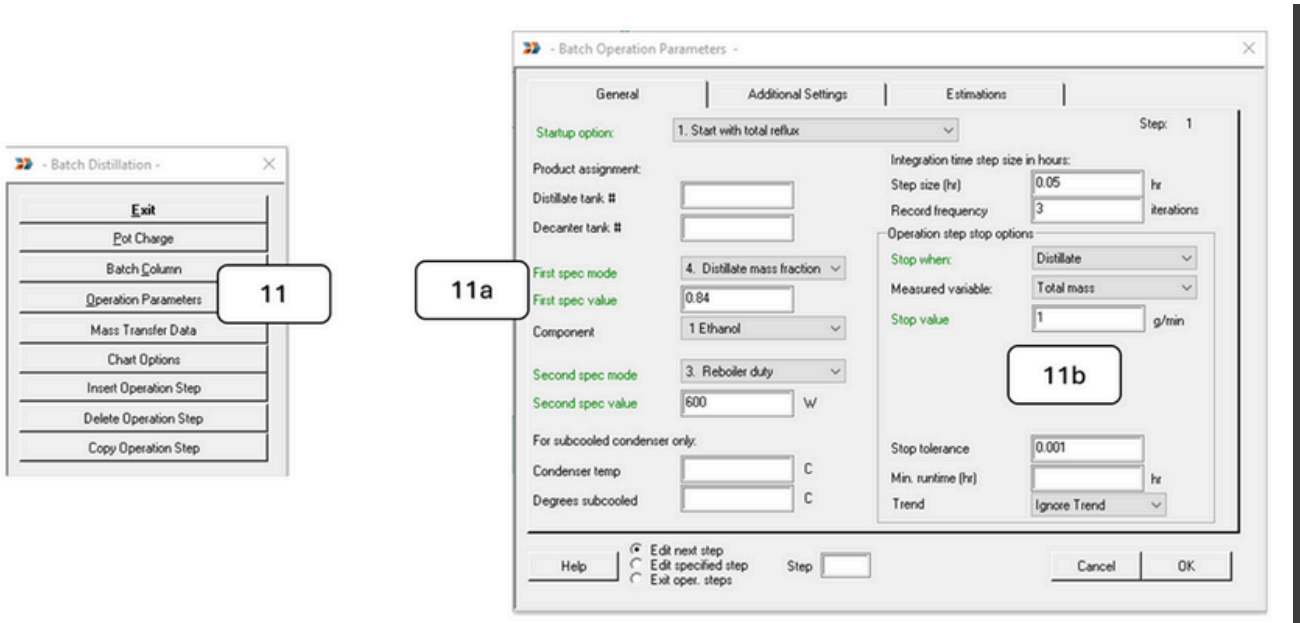


Figure 12: First Feasibility Check - Steps 11a and 11b.

This calculation is numerically much more challenging, and the solver needs a bit of help. Here we can get a converging simulation by allowing the program to use the starting values from the previous step (total reflux), see Figure 13.

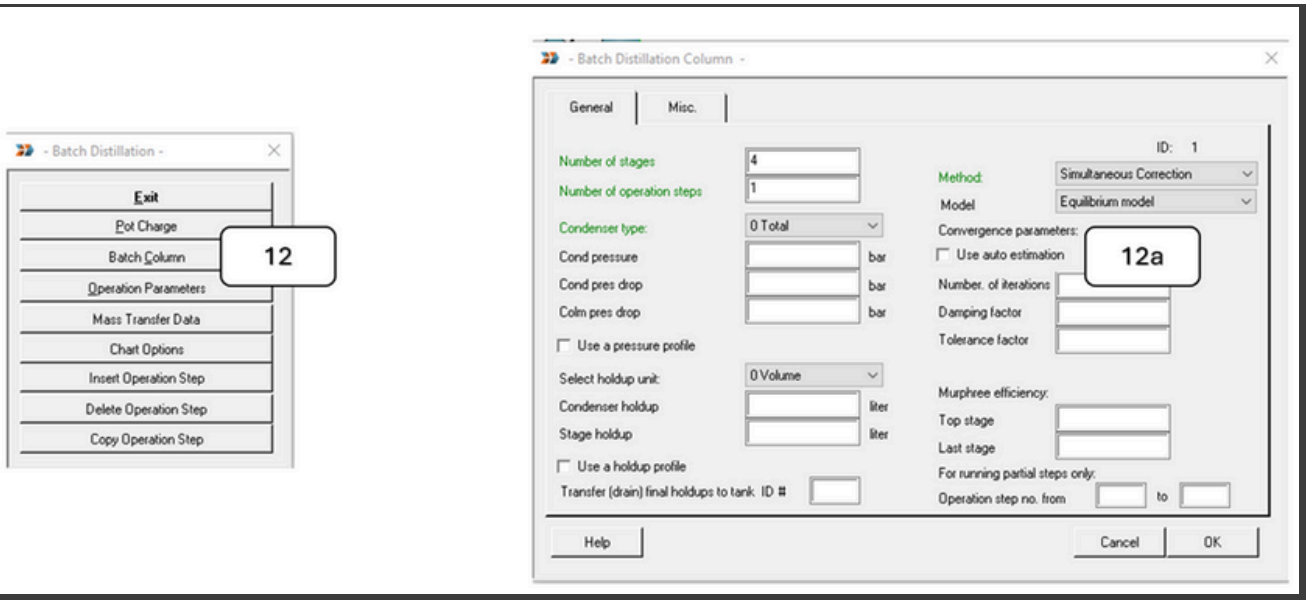


Figure 13: First Feasibility Check - Step 12.

Remove the checkmark in the box “Use auto estimation.”

Now we can Run the flowsheet.

The plot of distillate concentrations is of little interest, except for identifying the time when the stopping criteria are met. This happens at 63 minutes. At this time, around 18% of the ethanol in the pot has been recovered.

It is clear that running the distillation further would not make sense because the reflux ratio to reach the 84% is already above 100 and the distillate flow is approaching zero. It is recommended to use the different graphical reports to check out these (and other) results.

These two scenarios give a first impression of what can be reached with an idealized column containing four theoretical stages. The main results are summarized in Table 2.

Table 2: Main results of the first feasibility check.

	Constant reflux ratio of 10	Constant ethanol concentration
Operation time	**1 hour**	**1 hour**
Ethanol recovery	**54%**	**18%**
Ethanol purity	**80 – 83 wt.%**	**84 wt.%**

Using this model of the batch distillation plant and a model for the next operational step (adsorption), optimal operation strategies can be derived. However, this model relies on several assumptions that have not yet been experimentally validated, which could significantly alter the derived operation strategies.

For this first feasibility check, it was assumed that:

- the column has exactly four theoretical stages,
- no heat losses occur, and
- no liquid hold-up occurs, neither in the packing nor in the condenser.

The next section addresses these assumptions and shows how to get rid of them by finding the relevant parameters from experimental data.

Model Parameters Received from the Column Start-Up

In this section, measurements from the heat-up phase of the batch distillation plant are utilized to obtain parameters necessary for a more realistic simulation model compared to the one described previously. This section is intended for more experienced users; therefore, the specific steps taken in CHEMCAD are not explained in as much detail as previous sections of this paper.

Measured Data

The signals obtained during the start-up of the plant are depicted in Figure 14. A heat duty of 500 Watt is added to the pot and the valve in the condenser is set to total reflux. The condenser duty is calculated from the cooling water flowrate and it's temperature difference.

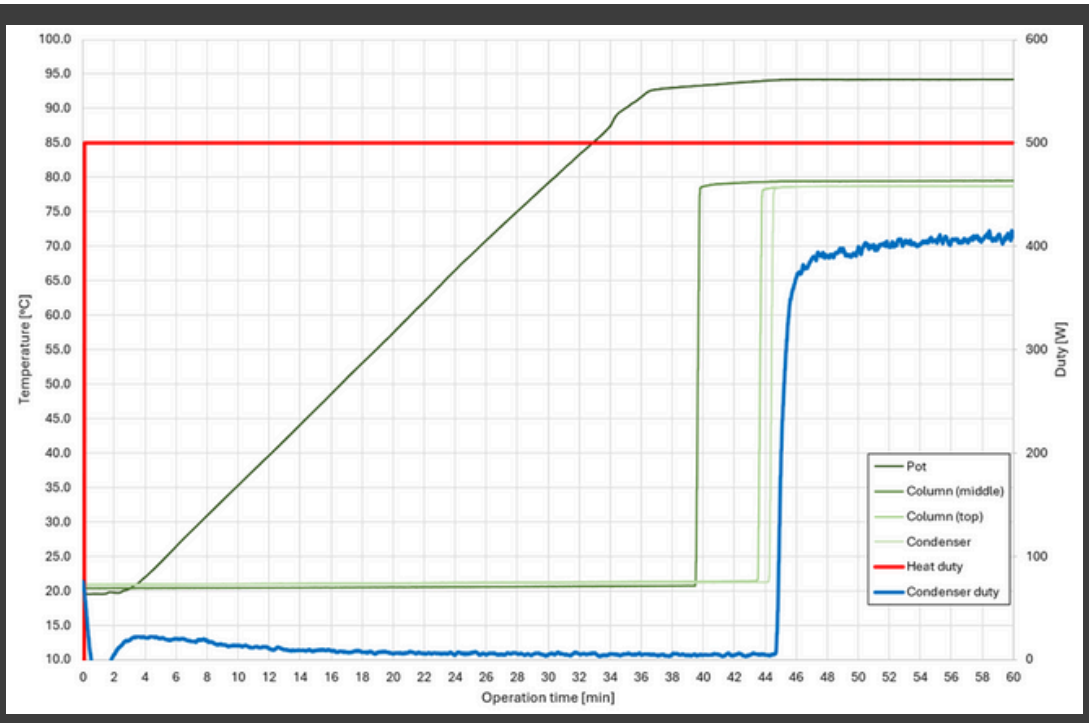


Figure 14: Temperature signals, heat duty, and condenser duty (calculated from flow rate and temperature difference) during the start-up phase of the batch distillation plant.

It takes some minutes until the heat mantel and glass of the pot are heated. After that, the temperature in the pot is increasing almost linearly. After 34 minutes of operation time the pot temperature suddenly increases and then drops again. This happens, because the mixture starts to boil, but there is still subcooled liquid in the pot. The occurring bubbles result in a better mixing of the liquid in the pot and the swinging in the temperature signal that was caused by inhomogeneities disappears. The temperature again increases linearly, but with a decreased slope. Liquid from the pot is evaporated and transported as vapor into the column. The vapor reaches the first temperature measurement in the column after approximately 40 minutes. Five minutes later, the vapor arrives in the condenser and the cooling water starts to remove heat. After one hour all profiles are flat and the steady state is reached.

Conclusions on the Initial Composition

The temperature profile in the pot contains information about the initial composition of the mixture in the pot. After the liquid heating and the occurrence of the first bubbles, the pot temperature returns to a linear profile at a temperature of 92.5 °C. This is the boiling temperature of the initial mixture. The bubble line of an ethanol water mixture at one bar is shown in Figure 15. The so received ethanol content in the mixture of approximately 8 weight percent was confirmed by a concentration measurement with a refractometer.

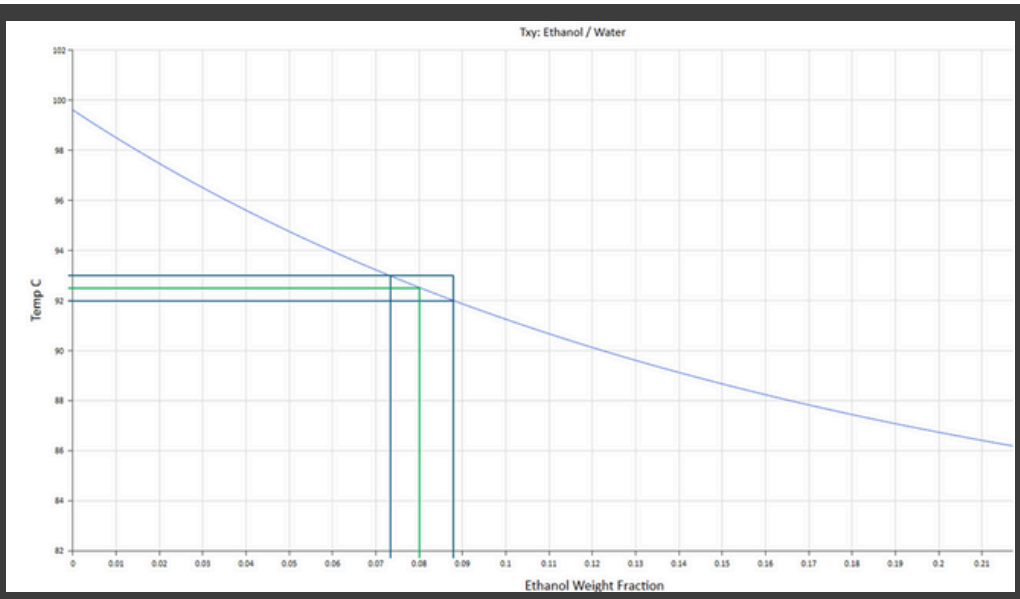


Figure 15: Bubble line of an ethanol water mixture at 1 bar and concentration estimation based on the temperature.

Conclusions on the Heat Losses of the Pot

With the known initial composition, the density and the mean heat capacity in the temperature range from 20°C to 90°C can be estimated. During the heat-up the slope of the pot temperature curve can be calculated with the energy balance for a closed system:

$$\frac{dT}{dt} \approx \frac{\dot{Q}}{m \cdot c}$$

This gives a theoretical increase of 2.4 Kelvin per minute. The experimental data shows an increase of 2.2 Kelvin per minute. Therefore, it can be concluded that approximately eight percent of the heat added are lost. From now on, the heat added to the pot is reduced accordingly in the simulations. For this case, this means that instead of the 500 Watt only 460 Watt are used in the model.

Conclusions on the Heat Losses of the Overall Plant

The difference in the heat duty added to the pot and the duty removed by the condenser gives the overall heat losses in the steady state total reflux case. This value is around 90 Watt, see Figure 14. A part (40 Watt) is lost directly at the heat mantel, so the remaining 50 Watt are emitted from the column and the condenser. CHEMCAD allows to consider heat losses depending on the temperature inside the column by specifying the heat transfer area, the heat transfer coefficient, and the ambient temperature:

$$\dot{Q}_{Loss} = U \cdot A \cdot \Delta T$$

For a first estimate we take a mean column temperature of 85°C, an ambient temperature of 20°C, and the inner column area of 0.19 m^2. This gives a heat transfer coefficient of $U = 4 \frac{W}{m^2 K}$.

The heat losses at the condenser and the separation chamber are lumped into this value.

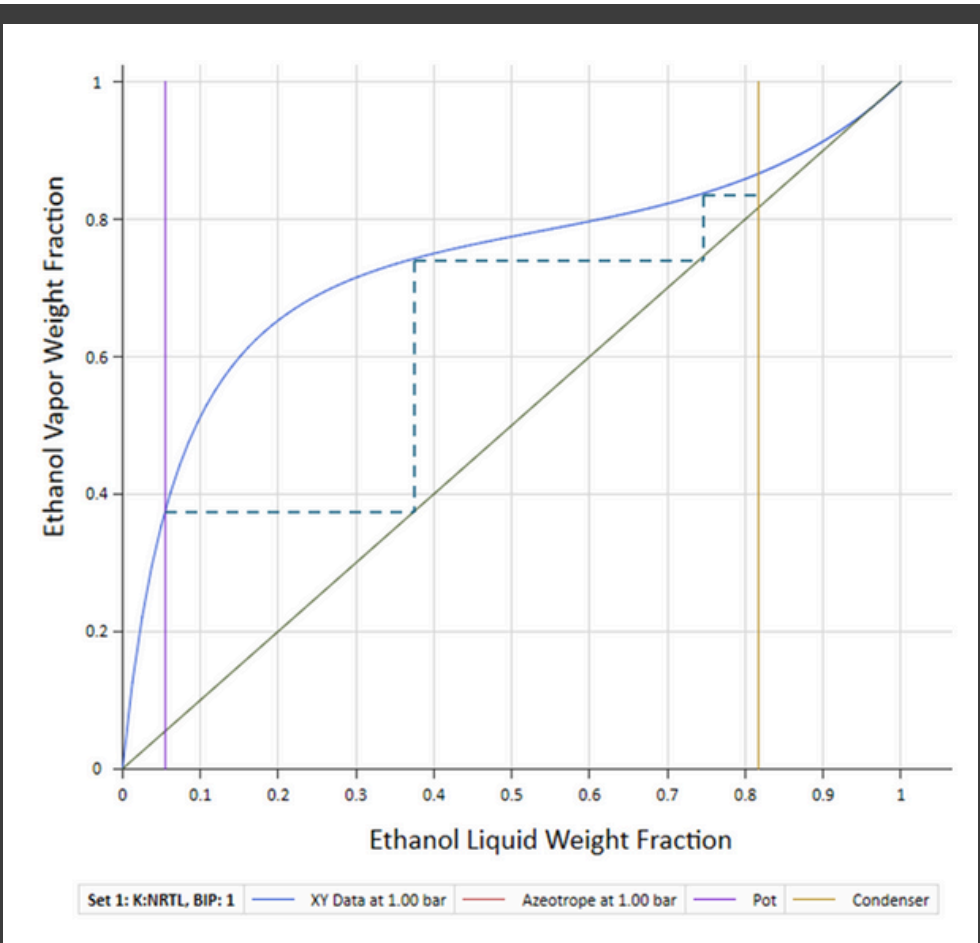


Figure 16: McCabe-Thiele plot based on the concentrations derived from measured temperatures.

Conclusions on the Number of Theoretical Plates

The composition in the condenser can be calculated from the temperature (78.7°C -> 81.7 wt.%), just as we did it to get the initial composition in the pot (Figure 15). The bubble line becomes quite flat at these high concentrations of ethanol. Therefore, it is recommended to confirm this concentration with an additional measurement. To estimate the number of theoretical plates, we also need the composition in the pot at steady state, which again can be read from the temperature (94.3°C -> 5.5 wt.%). These values are used in Figure 16 to estimate the number of theoretical plates in the column with the McCabe-Thiele method. Apparently less than three theoretical plates are found.

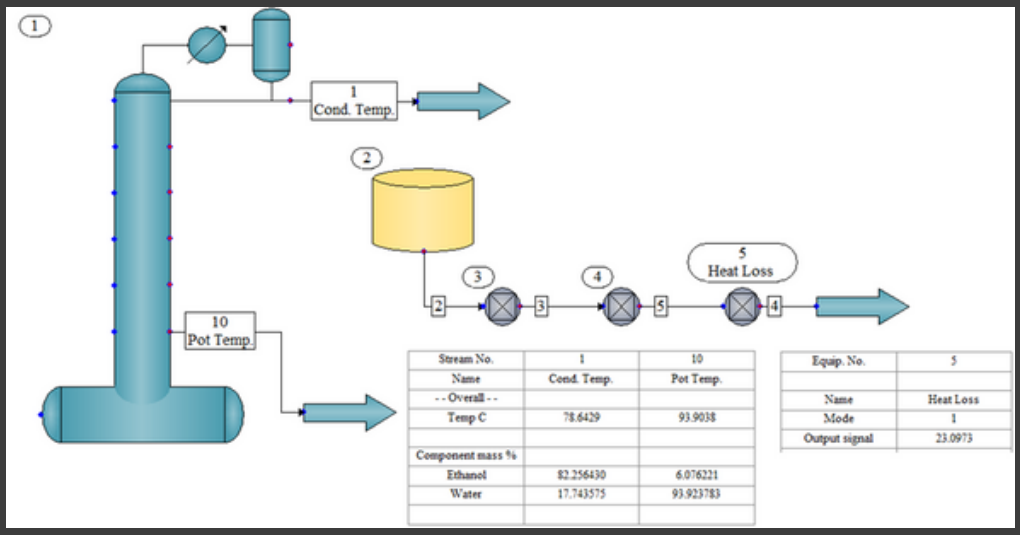
A non-integer number of plates can be handled in CHEMCAD by specifying a Murphree efficiency. We will keep calculating with four theoretical plates and adjust the efficiency of the plates to fit the measured data. As initial guess an efficiency of 1 is assumed in the pot and an efficiency of 0.8 in the condenser. Values in between are interpolated linearly.

Conclusions on the Liquid Hold-Ups

The change in temperature of the pot after reaching the bubble points is a clear indicator for a change of composition, which indicates that liquid is removed from the pot and stored in the columns packing and in the condenser. However, the composition of the removed liquid depends on the separation strength of the column, or the number of theoretical plates respectively. In columns with a higher separation strength, more ethanol is removed from the pot than in columns with little separation strength, where the water concentration in the upper parts of the column and in the condenser is higher. Therefore, the estimation of the liquid hold-ups can only be done together with the estimation of the number of theoretical plates, i.e. the Murphree efficiency.

Model for Parameter Regression

We resume to the model for the first feasibility check and add a distillate stream (Stream 1), a batch tank for the distillate (Unit 2), some controllers to calculate the heat loss (Units 3-5), and a side draw stream (Stream 10) to access the pot temperature.



Stream No.	1	10
Name	Cond. Temp.	Pot Temp.
- - Overall - -		
Temp C	78.6429	93.9038
Component mass %		
Ethanol	82.256430	6.076221
Water	17.743575	93.923783

Equip. No.	5
Name	Heat Loss
Mode	1
Output signal	23.0973

Figure 17: Flowsheet for model parameter regression.

Stream and UnitOp boxes are used to show the measurements to regress on the flowsheet (heat loss, pot, and condenser temperature. The result is shown in Figure 17.

Calculation of the Heat Loss

The heat loss is hidden in the heat duties calculated in the batch column. To access it, controller 3 reads the supplied heat duty during step 1, controller 4 the condenser duty removed during this step, and controller 5 calculates the difference. The engineering units are converted to give the heat loss in Watt. The individual controller specification screens are depicted in Figure 18. Alternatively, the Excel Data Map or a VBA UnitOp in CHEMCAD could have been used for these calculations.

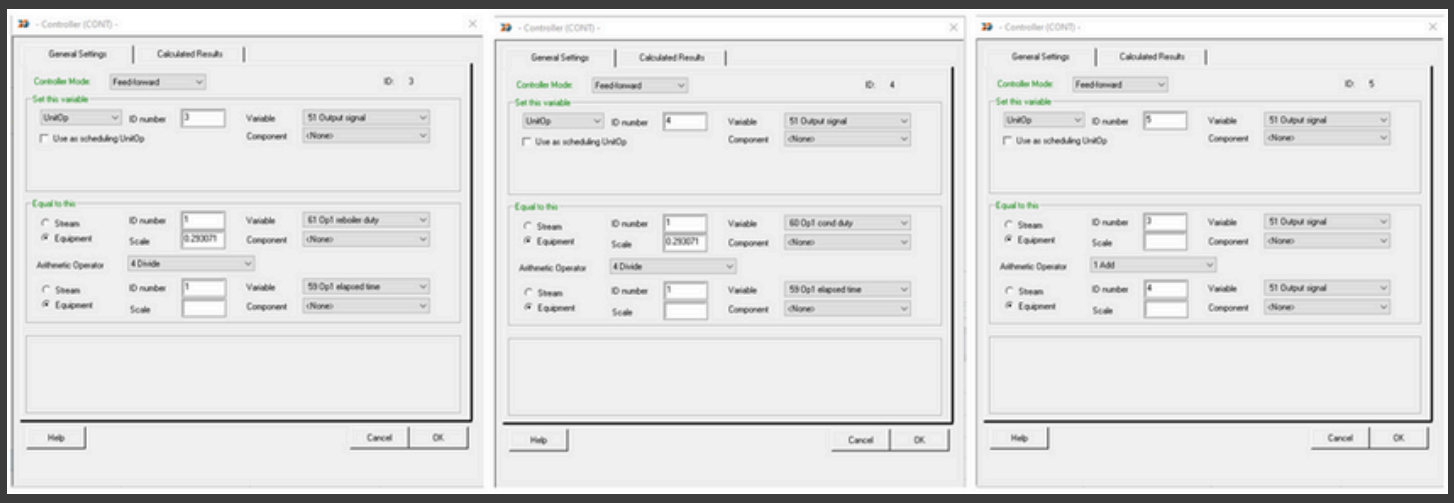


Figure 18: Controller specifications to calculate the heat loss in controller 5.

Hold-up and Efficiency Specifications

Two different liquid hold-ups can be specified in the batch column menu, the hold-up per stage and the hold-up in the condenser (Figure 19).

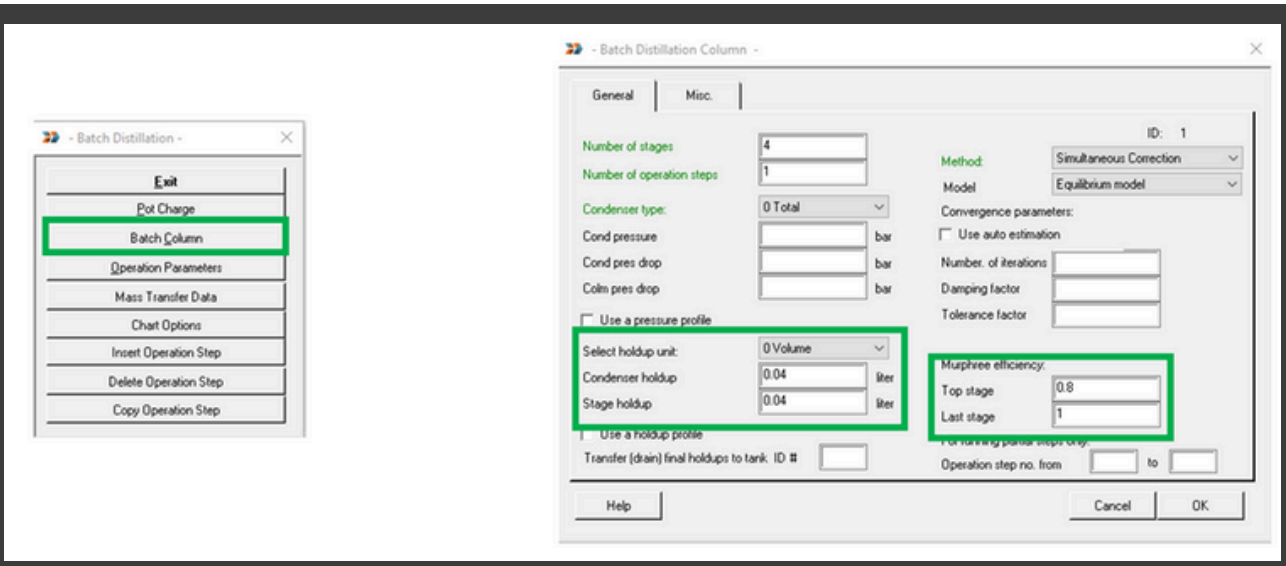


Figure 19: Hold-up and efficiency specifications.

From the measured data it is impossible to distinguish between these two hold-up volumes. Using empirical hold-up specification formulas, a value of approximately 4% per volume of packing is estimated. Spread over two stages this gives 0.04 liter per stage. The same value is used as initial value for the condenser hold-up. Neither is it possible to distinguish between the efficiencies of the individual stages at this moment. The efficiency of the pot (last stage) is therefore set to unity, for the top stage we set the initial value to 0.8. CHEMCAD interpolates linearly between these two values.

Heat Loss and Side Stream Specifications

The heat transfer to ambient and the stage for the side stream are specified in the Miscellaneous tab of the Batch Column menu, see Figure 20. We draw the side stream from the pot, i.e. tray four of four. The area of the column is divided by the number of stages (four) to get the area per tray.

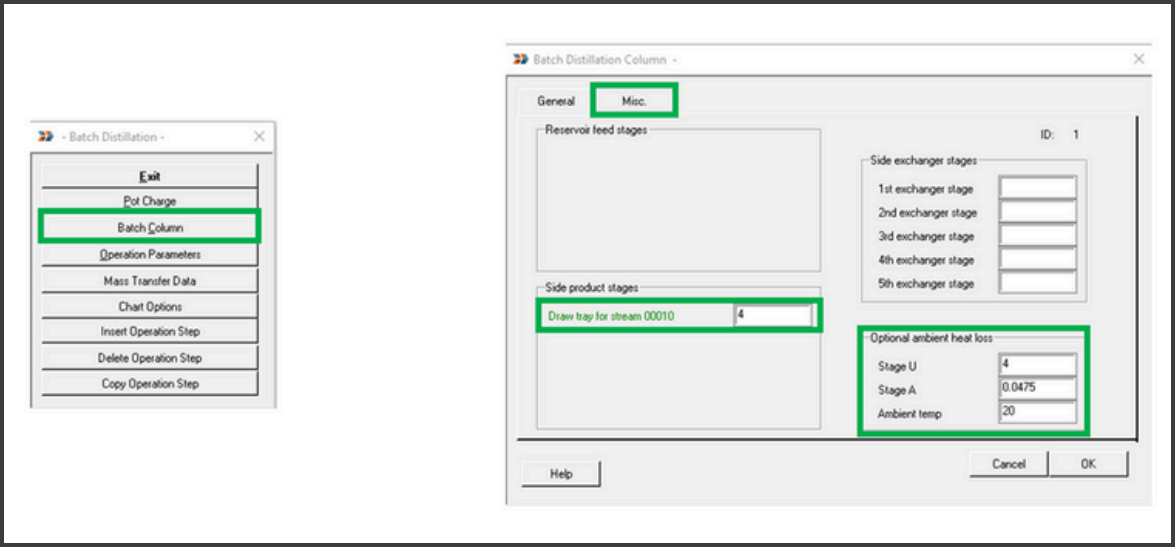


Figure 20: Heat loss and side stream specifications.

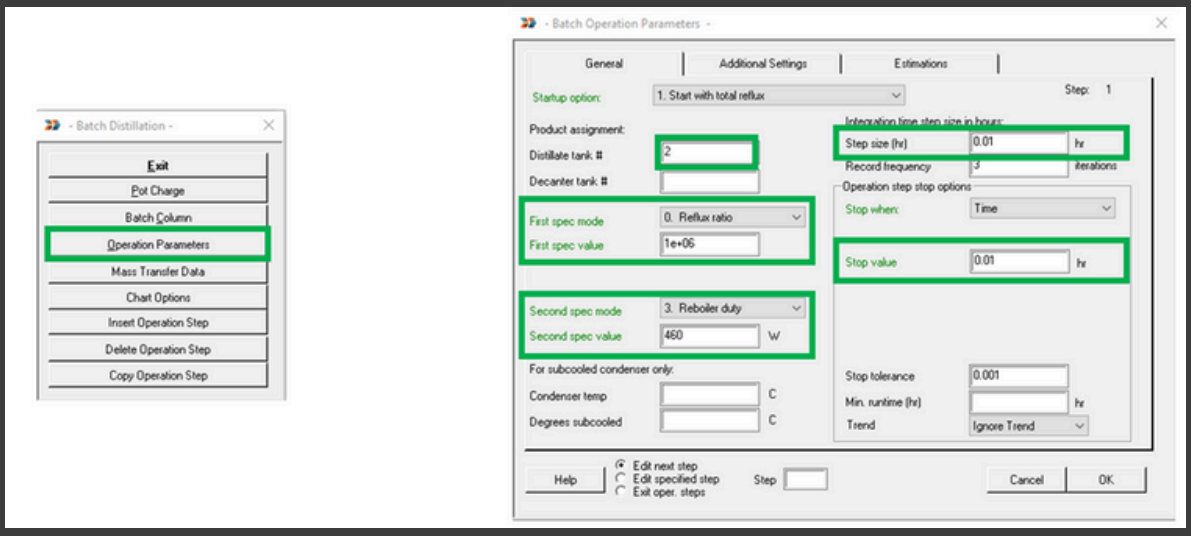


Figure 21: Operation step with total reflux.

Operation Step with Total Reflux

CHEMCAD requires at least one operation step to run the simulation. Therefore, a total reflux operation step lasting one time step (reduced to 36 sec / 0.01 hr) is defined, see Figure 21. The distillate (practically zero) is sent to tank 2.

Accessing the Pot Temperature

A new screen pops up after pressing the OK button of the operation parameter specification screen (Figure 22). This screen allows us to specify the side product stream. No product is drawn, the side stream is only used to read the pot temperature and composition. Specifying the numerical zero (1e-6) is required though to get the pot composition.

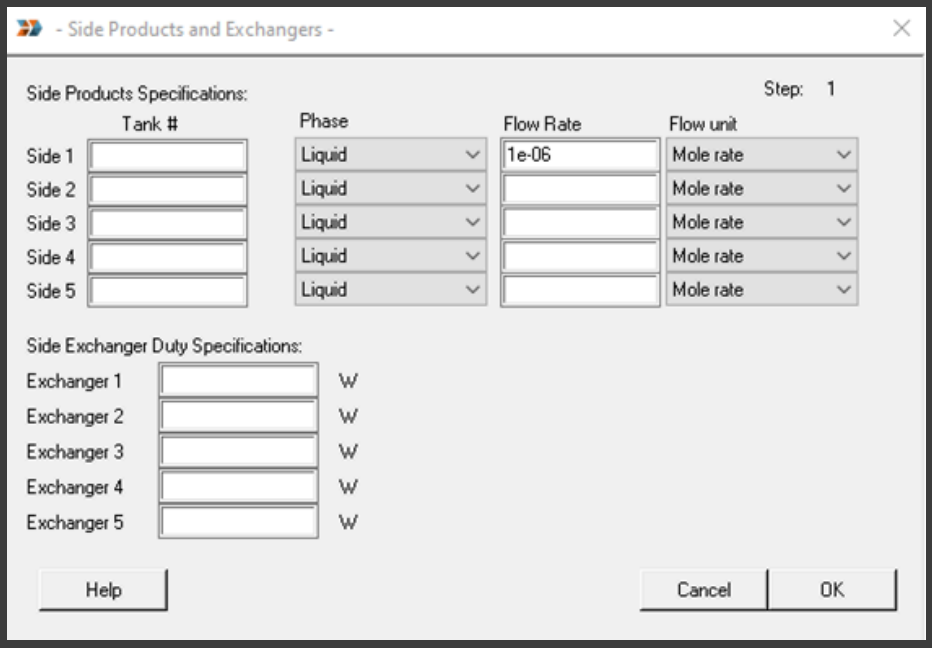


Figure 22: Side product specification.

Test Run

After running the simulation, the calculated values for the measured temperatures and the overall heat loss can be found in the data boxes, see Figure 17. The temperatures and the heat loss are already in the range of the measured values, but there is still space for improvement.

Simultaneous Parameter Regression

The three remaining model parameters, namely condenser hold-up, heat transfer coefficient, and top stage efficiency are correlated and must be regressed simultaneously. This is done with the Data Reconciliation tool in CHEMCAD.

The measured values are stored in an Excel table accessed via Data Map, see Figure 23. Figure 24, Figure 25, and Figure 26 show how the data reconciliation is set up. The objective function of the data reconciliation problem is full of local minima. To get better results, the number of restarts is increased to 200. Print level 4 gives a more detailed report on the performance of the optimization. All values are reached within the measurement tolerances. The resulting model parameters are a condenser hold-up of 62 ml, a heat transfer coefficient of 8.7 W/m^{2}K, and a top stage efficiency of 82 %.

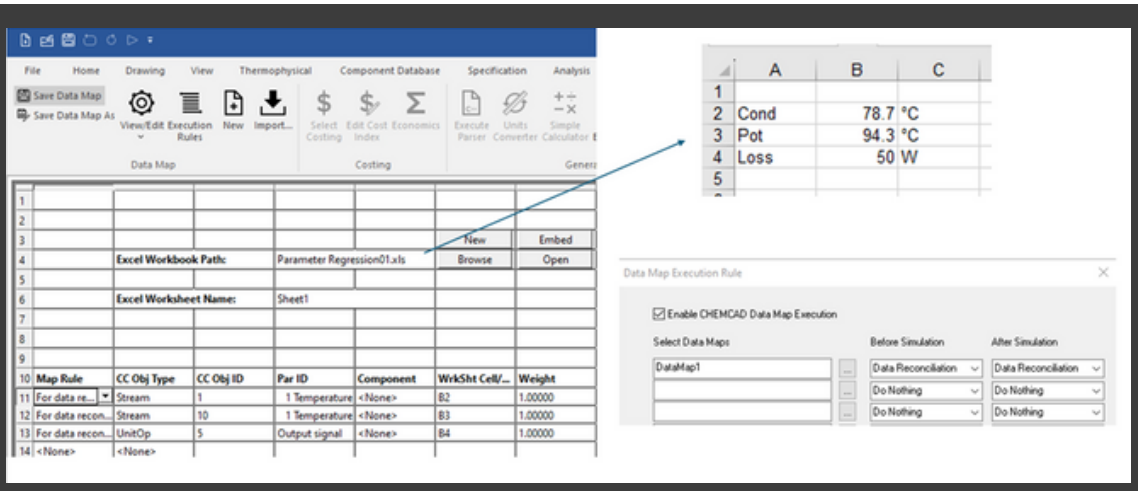


Figure 23: Data Map settings for the Data Reconciliation.

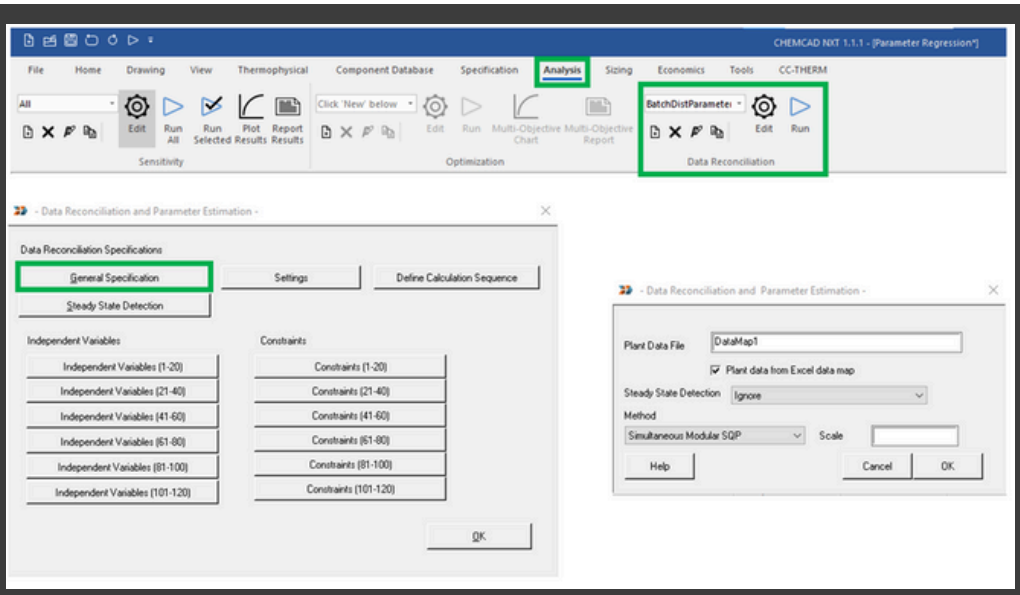


Figure 24: Link to the measured data and selection of the optimizer.

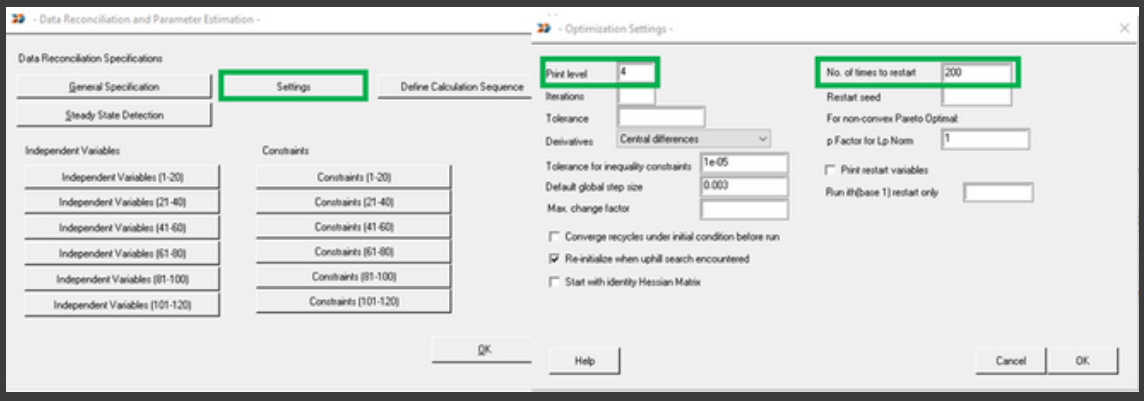


Figure 25: Detailed settings for the optimizer.

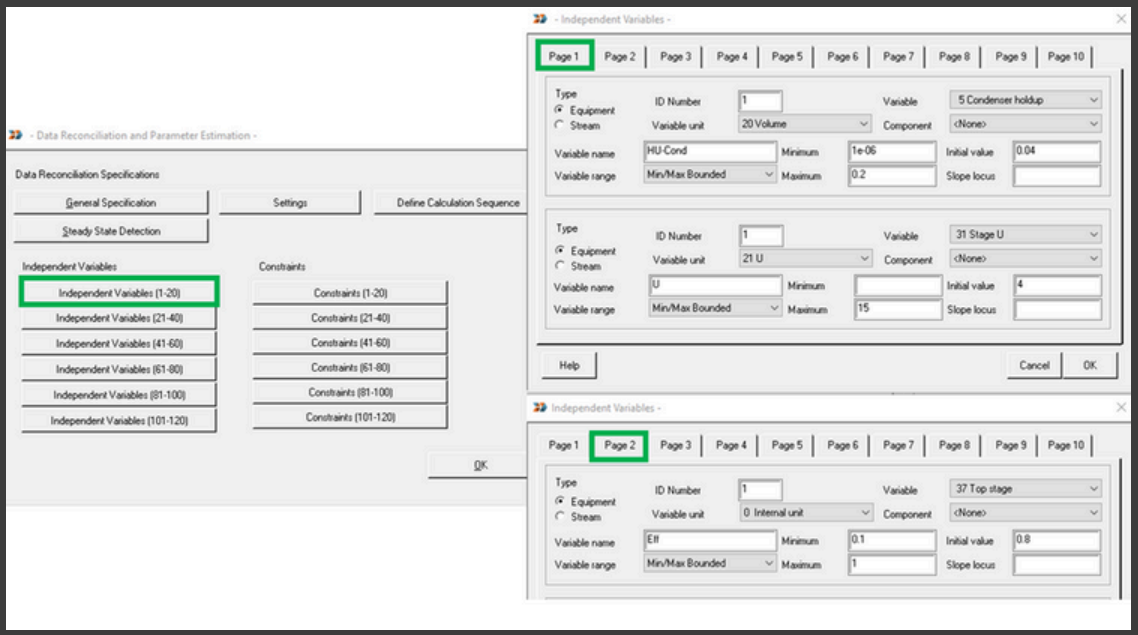


Figure 26: Specification of the independent variables.

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Simultaneous Modular SQP Final Results:

Independent Variables in User Specified Engineering Units:

                                Estimation       Final Value
HU-Cond              X[  0] =   4.000000e-02     6.226845e-02
U                    X[  1] =   4.000000e+00     8.660980e+00
Eff                  X[  2] =   8.000000e-01     8.157570e-01

Objective Function              Initial Value    Final Value
                                 3.586511e-02     9.293255e-06

Inequality Constraints                  Initial Value    Final Value
HU-Cond-LB                 g[  0] =     1.412551e-03     2.198954e-03
UB-HU-Cond                 g[  1] =     5.650347e-03     4.863944e-03
U-LB                       g[  2] =     7.044379e-01     1.525281e+00
UB-U                       g[  3] =     1.937204e+00     1.116362e+00
Eff-LB                     g[  4] =     7.000000e-01     7.157570e-01
UB-Eff                     g[  5] =     2.000000e-01     1.842430e-01

No. of restarts       = 200
Restart Seed          = 0
No. of SQP iterations = 1894
No. of function calls = 16138
No. of cores          = 3
Parallel computing    = 1
Max. iterations/thread = 100
Default step size =  0.00300
Max change factor =  0.00000
Central differences.

Input Data and Errors
No.  TagName                MeasuredValue  ReconciledValue  Weight  Error
   0 S_1_Temperature         7.87000e+01    7.86504e+01      1.00     1.41088e-03
   1 S_10_Temperature        9.43000e+01    9.42420e+01      1.00     1.57799e-03
   2 U_5_Output signal       5.00000e+01    5.03589e+01      1.00    -7.17834e-03
```

Figure 27: Results of the data reconciliation.

Model Prediction Accuracy

The measurements used for finding the model parameters are fitted almost perfectly. In the next step the accuracy of the model for predicting the operation of the batch distillation plant is evaluated. Therefore, distillate is drawn after 13 minutes of total reflux at a constant reflux ratio of 5. Two operation steps are added to the model built in the previous section: A step of total reflux lasting 0.2 hours and a step with a reflux ratio of 5 lasting 0.79 hours. This gives an overall simulation time of one hour. In Figure 28 the calculated and the measured condenser temperatures are compared. They are in a good agreement.

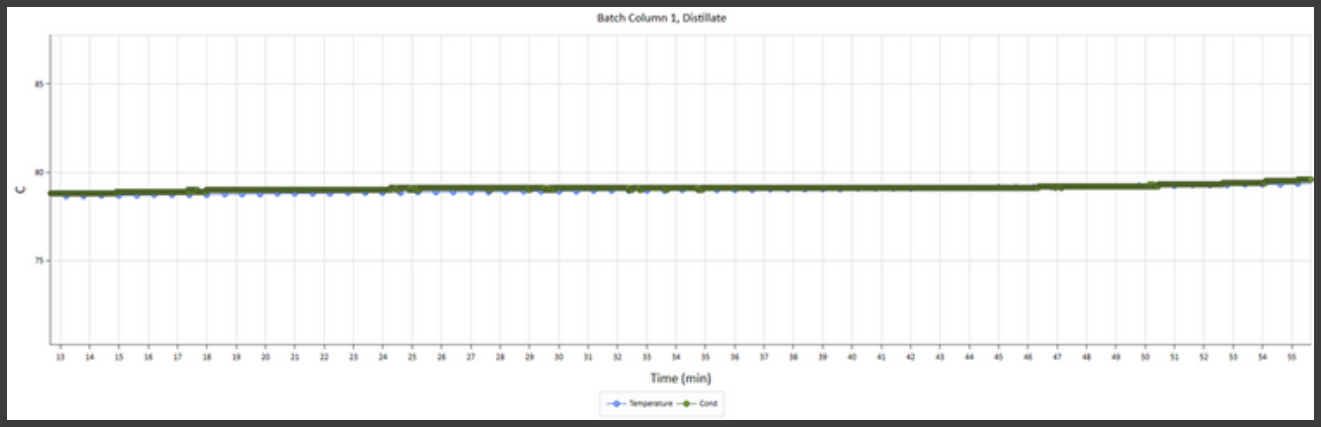
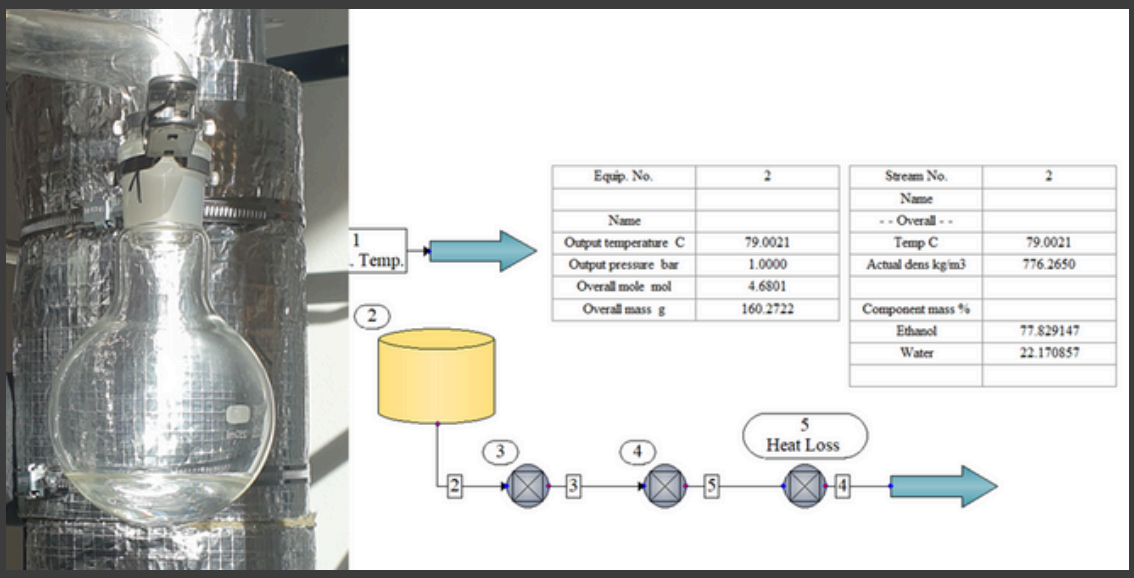


Figure 28: Comparison of calculated and measured condenser temperatures using a reflux ratio of 5.

One receiver tank (~200 ml) is filled with a mixture of 77 wt.% of Ethanol. This fits perfectly with the model results.



Equip. No.	2
Name	
Output temperature C	79.0021
Output pressure bar	1.0000
Overall mole mol	4.6801
Overall mass g	160.2722

Stream No.	2
Name	
- - Overall - -	
Temp C	79.0021
Actual dens kg/m3	776.2650
Component mass %	
Ethanol	77.829147
Water	22.170857

Figure 29: Real receiver tank and simulated receiver tank. The product concentration is verified by a gravimetric measurement.

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