

# A COMPREHENSIVE REAL-WORLD DISTILLATION EXPERIMENT

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**D**istillation is the major separation method in industry, and its analysis brings together many important chemical engineering principles: component balances, energy balances, heat transfer, and phase equilibrium. Distillation is a core subject in all chemical engineering educational programs. The basic concepts can be very effectively conveyed by simple graphical methods, particularly in binary systems where McCabe-Thiele diagrams clearly demonstrate the effects of number of stages, reflux ratio, relative volatility (pressure), and vapor-liquid phase nonideality. Many textbooks<sup>[2-7]</sup> covering distillation principles have appeared since the 1950 pioneering book by Robinson and Gilliland.<sup>[1]</sup>

The examples used in introductory courses usually make many simplifying assumptions in order to not cloud the fundamental concepts. These include theoretical trays, total condenser with saturated liquid reflux and distillate, partial reboiler, and a single feed stream (either saturated liquid or saturated vapor). Operating lines are represented as functions of the reflux ratio (reflux flow divided by distillate flow). University distillation experiments<sup>[8-12]</sup> are usually small scale with glass components, which are not typical of industrial distillation systems.

The experimental distillation column in the Lehigh University Process Laboratory has some features that are more representative of actual industrial columns, and these features complicate the analysis.

For many years, our senior students have found that performing experiments and analyzing the performance of this column is a very effective way to extend the basic concepts and theory that they learned in their junior-year mass transfer course.

This paper describes the equipment, startup procedure, reconciliation of the raw data, energy balances around various sections of the column, calculation of tray efficiencies, and simulation of the column to compare theoretical predictions with experimental data.

Safety issues are fairly minimal in the experiment. Methanol is flammable but is mixed with water except in the reflux drum. An alarm is activated if the flowrate of cooling water to the condenser is too low. Another alarm is activated if the base pressure gets too high, indicating column flooding. An interlock shuts off the feed pump if the base level gets too high.



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## PROCESS STUDIED

Figure 1 gives the flowsheet of the unit. The distillation column separates methanol-water at 1 atm pressure. It is 8 inches in diameter and has 14 trays with three Glitch V-1 ballast caps on each tray. Feed is pumped from a tank (cross-sectional area = 3.57 ft<sup>2</sup>) through a control valve and into the tube side of a heat exchanger where it is heated by the overhead vapor from the column. This setup is sometimes called an “economizer.” The feed is still subcooled when it is fed to one of three alternative trays (4, 6, or 8). Trays are numbered from the bottom.

Saturated steam at 35 psig flows through a control valve into the shell side of a vertical thermosiphon reboiler. Condensate leaves through a steam trap. The steam pressure in the shell of the reboiler is 5 psig. Vapor flows up through the column and enters the shell side of the feed preheater, which is a horizontal tube-in-shell heat exchanger. Any liquid that has condensed flows out the bottom of the shell side of the preheater into the reflux drum. Vapor leaves the top of the feed preheater and goes to the shell side of two more heat exchangers in series with cooling water flowing countercurrently through the tube side. The vapor is totally condensed and the liquid flows by gravity to the reflux drum. The shell side of the final condenser is vented to the atmosphere, so the pressure in the column is 1 atm and the reflux is subcooled.

Liquid from the reflux drum is pumped and split into a reflux stream and a distillate stream with control valves in

both lines setting the flowrates of the two streams. Reflux is fed on Tray 14 at the top of the column. Distillate goes to a distillate tank (cross-sectional area = 1.24 ft<sup>2</sup>).

The bottoms stream from the base of the column goes to a cooler and into a pump. The pump discharge goes through a control valve and into a bottoms tank (cross-sectional area = 1.24 ft<sup>2</sup>). Both the distillate and the bottoms tanks can be drained into the feed tank. These three tanks are used to determine flowrates by tank gauging during the run when steady-state conditions have been attained. All control valves are electronic and are manually positioned from five controller faceplates.

Instrumentation includes temperature measurements of all streams and on all trays. Base pressure, steam supply pressure, and steam pressure in the reboiler are measured. Level indicators and sight glasses are available on the reflux drum and the column base. Sight glasses are available on the feed, bottoms, and distillate tanks.

Liquid samples can be taken from the feed tank, from the line leaving the reflux drum, and from the base of the column. Hydrometers are used to measure the density, from which compositions can be calculated because of the significant difference in density between water (sp. gr. = 1) and methanol (sp. gr. = 0.792).

## STARTUP PROCEDURE

One of the most important educational features of the experiment is requiring the students to develop and perform a

safe and rapid startup from a cold column initial condition. After electric power is turned on, the first step is to open the cooling water inlet valve and to guarantee by visual inspection that there is sufficient cooling water flowing to the two condensers at the top of the column.

The feed pump is started and the feed control valve is opened to bring liquid into the base of the column. The feed pump is shut off and the feed valve closed when the liquid level in the column base is about 80% full as indicated by both the sight glass and the level transmitter. Then steam is slowly introduced into the shell side of the reboiler. It takes about 15 minutes to heat up the liquid in the column

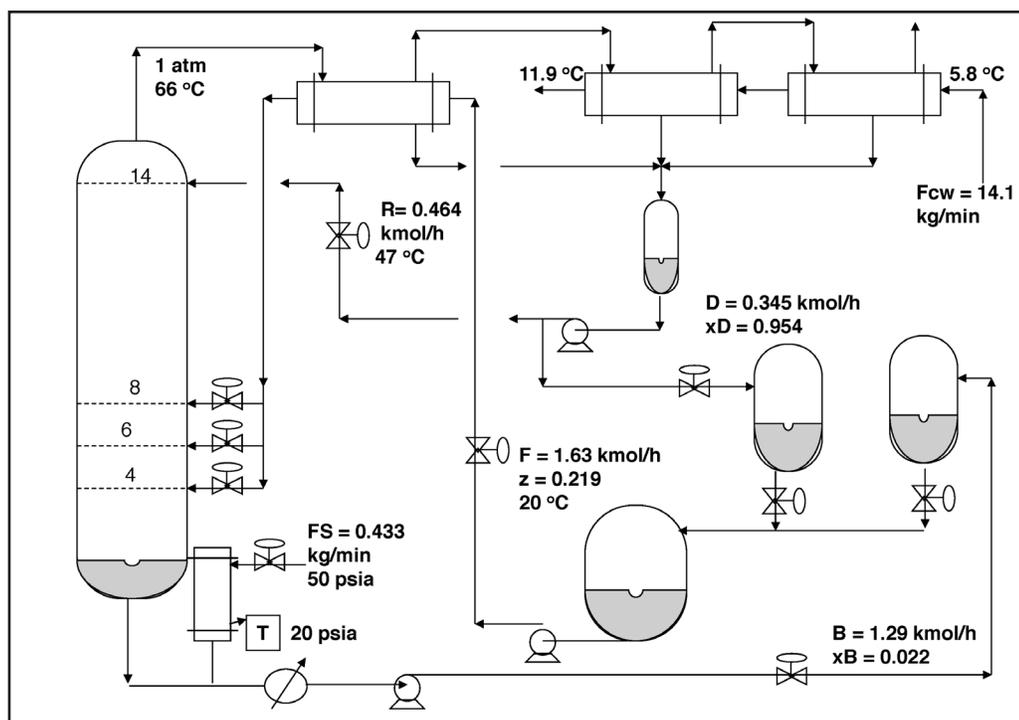


Figure 1. Flowsheet and experimental conditions.

base, at which point vapor begins to flow up the column. The rising hot vapor heats the metal of the trays and the vessel wall. The temperature on Tray 1 increases, then Tray 2, and so forth up the column. It takes about 10 more minutes for the hot vapor to work its way up to Tray 14.

When vapor starts to come overhead, the liquid level in the reflux drum begins to rise. When the drum is about 25% full, the overhead pump is started and the reflux valve is opened. The distillate valve is completely closed. The position of the reflux valve is adjusted to maintain a constant liquid level in the reflux drum.

Thus the initial part of the startup is putting the column on total reflux. The flowrates of the feed, distillate, and bottoms streams are all zero. The steam valve is in a fixed position and the reflux valve is controlling reflux drum level. The base level is uncontrolled, but there must be adequate liquid in the base. Material from the initial quantity charged to the column base goes to build liquid inventory on all the trays and in the reflux drum. If the base level gets too low during the startup, more feed is introduced.

Once reflux starts flowing down the column and contacting the rising vapor, fractionation begins. We will analyze the total reflux operating conditions in a later section of this paper. First we will examine normal steady-state operation with feed and product streams.

## NORMAL FEED RUN

The transition from total reflux to feed operations is achieved by starting the feed pump and opening the feed valve to about 40%. The steam valve is set at about 50%. The distillate valve is opened to about 15% and a level controller is put on automatic to adjust the distillate valve to maintain the liquid level in the reflux drum. The bottoms pump is started and the bottoms valve is opened to about 25%. A second level controller is put on automatic to adjust the bottoms valve to maintain the liquid level in the base of the column.

### Raw data

It takes about 45 minutes for the column to come to a steady-state condition as indicated by constant temperatures and levels. During this period the bottoms and distillate tanks are draining back into the feed tank. When the valves in the exit lines of these product tanks are closed, the level in the feed tank falls and the levels in the product tanks climb. Recording level changes over a period of time gives flowrate information for feed F, distillate D, and bottoms B. This “tank gauging” procedure is what would often be done in industry using feed and product tank level changes over a prolonged test period. Cooling water and steam condensate flowrates are determined by the “bucket and stopwatch” method (ice is used in the bucket to catch the hot steam condensate).

Liquid samples are taken of the feed, reflux (distillate), and

bottoms in a graduated cylinder, and hydrometers are used to find densities, from which compositions can be calculated. Temperatures of all streams and on all trays are recorded. Table 1 gives some of the important raw data from a typical run.

The flowrate of the reflux cannot be determined during the run, but it can be obtained after the column has been shut down. Liquid is retained in the reflux drum. The overhead pump is started and the reflux valve is set in the same position that it was during the run. Nothing is entering the reflux drum, so the drop in level with time gives the reflux flowrate that we had during the run.

### Data reconciliation

A major educational feature of the experiment is exposing the students to real data with its inherent inaccuracies. Flow and composition measurements are imprecise in an industrial environment. Material balance calculations show that “In” is not equal to “Out” in terms of both mass (and molar flowrates in this non-reactive process where moles are conserved) and components. The students are forced to develop a rational data reconciliation procedure to make the total molar and component balances perfect before getting into further analysis to generate McCabe-Thiele diagrams, determine tray efficiencies, and check energy balances.

The data given in Table 1 are used to calculate molar compositions and molar flowrates. The first and second columns in

	sp. gr.	Flowrate
Feed	0.927	677 cm <sup>3</sup> /min
Distillate	0.797	226 cm <sup>3</sup> /min
Bottoms	0.992	396 cm <sup>3</sup> /min
Reflux	0.797	304 cm <sup>3</sup> /min
Steam		0.433 kg/min
CW		14.1 kg/min
	Temperatures (°C)	
Feed Tank	20	
Feed to Column	63	
CW Into Condenser	5.8	
CW Out Condenser	11.9	
Reflux Drum	47	
Tray 1	89	
Tray 14	66	

The specific gravity of methanol is 0.7815 and that of water is 1. The molecular weights are 32 and 18, respectively.

*One possible data reconciliation procedure is to assume that the flowrates of the distillate and bottoms are correct and that the feed flowrate is the sum of the two product flowrates. This makes the total molar balance perfect.*

	Calculated Molar Flowrate (kmol/h)	Calculated Mole Fraction Methanol	Adjusted Molar Flowrate (kmol/h)	Adjusted Mole Fraction Methanol
Feed	2.056	0.2322	1.631	0.2191
Distillate	0.345	0.954	0.345	0.954
Bottoms	1.286	0.022	1.286	0.022
Reflux	0.464	0.954	0.464	0.954

Table 2 give the calculated molar flowrates of the feed F, bottoms B, the distillate D, and the reflux R, together with the molar compositions of these streams ( $z$ ,  $x_B$ , and  $x_D$ ). Ideal mixing of methanol and water is assumed.

Adding D and B gives 1.621 kmol/h, which is not equal to F (2.056 kmol/h). Do we have a leak in the equipment? No, the flow measurements are not precise. In addition, the feed tank has an internal heat exchanger of unknown size, which occupies a portion of the cross-sectional area. So the tank gauging of the feed would be expected to give calculated flowrates that are too high.

Therefore one possible data reconciliation procedure is to assume that the flowrates of the distillate and bottoms are correct and that the feed flowrate is the sum of the two product flowrates. This makes the total molar balance perfect. The adjusted feed flowrate is  $F_{adj}$ .

To make the methanol component balance perfect, the feed composition  $z$  is back calculated assuming the distillate and bottoms compositions and flowrates are correct. The adjusted feed composition is  $z_{adj}$ .

$$F_{adj} = D + B = 0.345 + 1.286 = 1.631 \text{ kmol/h}$$

$$z_{adj} = \frac{Dx_D + Bx_B}{F_{adj}} = \frac{(0.345)(0.954) + (1.286)(0.022)}{1.631} = 0.2191 \quad (1)$$

The experimental value of  $z$  is 0.232, and the adjusted  $z_{adj}$  is 0.219, so there is only a minor adjustment. The last two columns in Table 2 give the flowrates and compositions used in the subsequent analysis.

### McCabe-Thiele diagram

Figure 2 gives the McCabe-Thiele diagram using a rectifying operating line (ROL) with a slope that considers the effect of subcooled reflux. The slope of an operating line in any section of the column is the ratio of the liquid to vapor flowrates in that section. The flowrate of the subcooled reflux is 0.464 kmol/h and its composition is 0.954 mole fraction methanol. It is not at its bubblepoint temperature but is subcooled at 46.9 °C while the temperature on Tray 14 is 66.3 °C. An energy balance is used to calculate the internal liquid flowrate LR in the rectifying section of the column.

$$LR = R \left[ 1 + \frac{C_p (T_{14} - T_R)}{\Delta H_v} \right]$$

$$= (0.464 \text{ kmol/hr}) \left[ 1 + \frac{(80.8 \text{ kJ/kmol-K})(66.3 - 46.9)}{36,200 \text{ kJ/kmol}} \right] = 0.484 \text{ kmol/hr} \quad (2)$$

$$LR/VR = \frac{LR}{LR + D} = \frac{0.484}{0.484 + 0.345} = 0.584$$

Liquid heat capacity is calculated using the molar composition and the component liquid molar heat capacities (methanol = 81.03 kJ kmol<sup>-1</sup> K<sup>-1</sup> and water = 74.93 kJ kmol<sup>-1</sup> K<sup>-1</sup>). Heat of vaporization is calculated using molar composition and component heats of vaporization (methanol = 36,000 kJ/kmol and water = 40,860 kJ/kmol). In later calculations of enthalpy, vapor molar heat capacities are used (methanol = 49.28 kJ kmol<sup>-1</sup> K<sup>-1</sup> and water = 36.0 kJ kmol<sup>-1</sup> K<sup>-1</sup>).

A similar energy balance around the feed tray gives a  $q$  value of 1.035 because the subcooled feed temperature is 62.6 °C while the temperature on the feed tray (Tray 4) is 81.1 °C. Now the operating lines can be drawn.

A value for Murphree vapor-phase efficiency is guessed, and stages are stepped off between the stripping operating line (SOL) and a fraction of the vertical distance to the vapor-liquid equilibrium line, starting at the

bottoms composition  $x_B$ . The partial reboiler is assumed to be an equilibrium stage. Aspen NRTL physical properties are used to generate the VLE curve at 1 atm.

Unlike a design problem in which the optimum feed tray occurs at the intersection of the ROL and SOL, the analysis of an operating column has a fixed feed stage. So we must step back and forth between the SOL and the VLE curve until we reach Tray 4. Then we can step to the ROL.

By trial and error, an efficiency of 65 % was found to give a vapor composition leaving Tray 14 that is equal to the distillate composition  $x_D = 0.954$ . See Figure 2.

The experimental temperature profile (the circles) is shown in Figure 3 (following page). The solid line is the predicted temperature profile from a simulation discussed later in this paper.

### Energy balances

Energy balances can be made around various sections of the column. The heat removed in the three overhead heat exchangers condenses and subcools the vapor leaving the top of the column. The flowrate of this vapor is the sum of the flowrates of the reflux and the distillate. The heat transferred into the cooling water in the condenser is:

$$\begin{aligned}
 QCW &= FCW * C_p * (T_{out} - T_{in}) \\
 &= (14.1 \text{ kg/min})(4.163 \text{ kJ/kg} \cdot \text{K})(11.92 - 5.85) (\text{min}/60 \text{ sec}) = 5.94 \text{ kW} \quad (3)
 \end{aligned}$$

*Unlike a design problem in which the optimum feed tray occurs at the intersection of the ROL and SOL, the analysis of an operating column has a fixed feed stage. So we must step back and forth between the SOL and the VLE curve until we reach Tray 4. Then we can step to the ROL.*

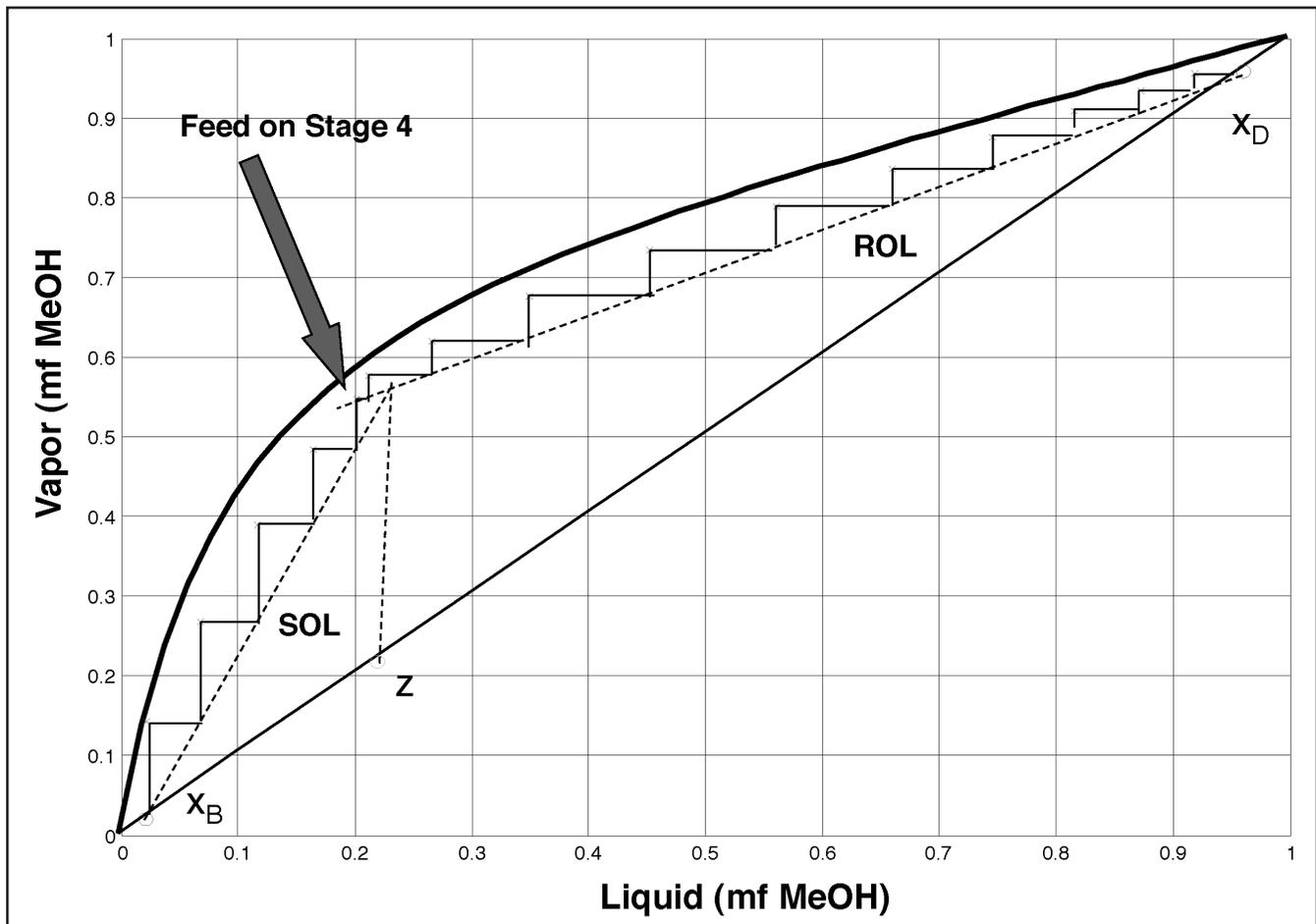


Figure 2. McCabe-Thiele diagram; feed run.

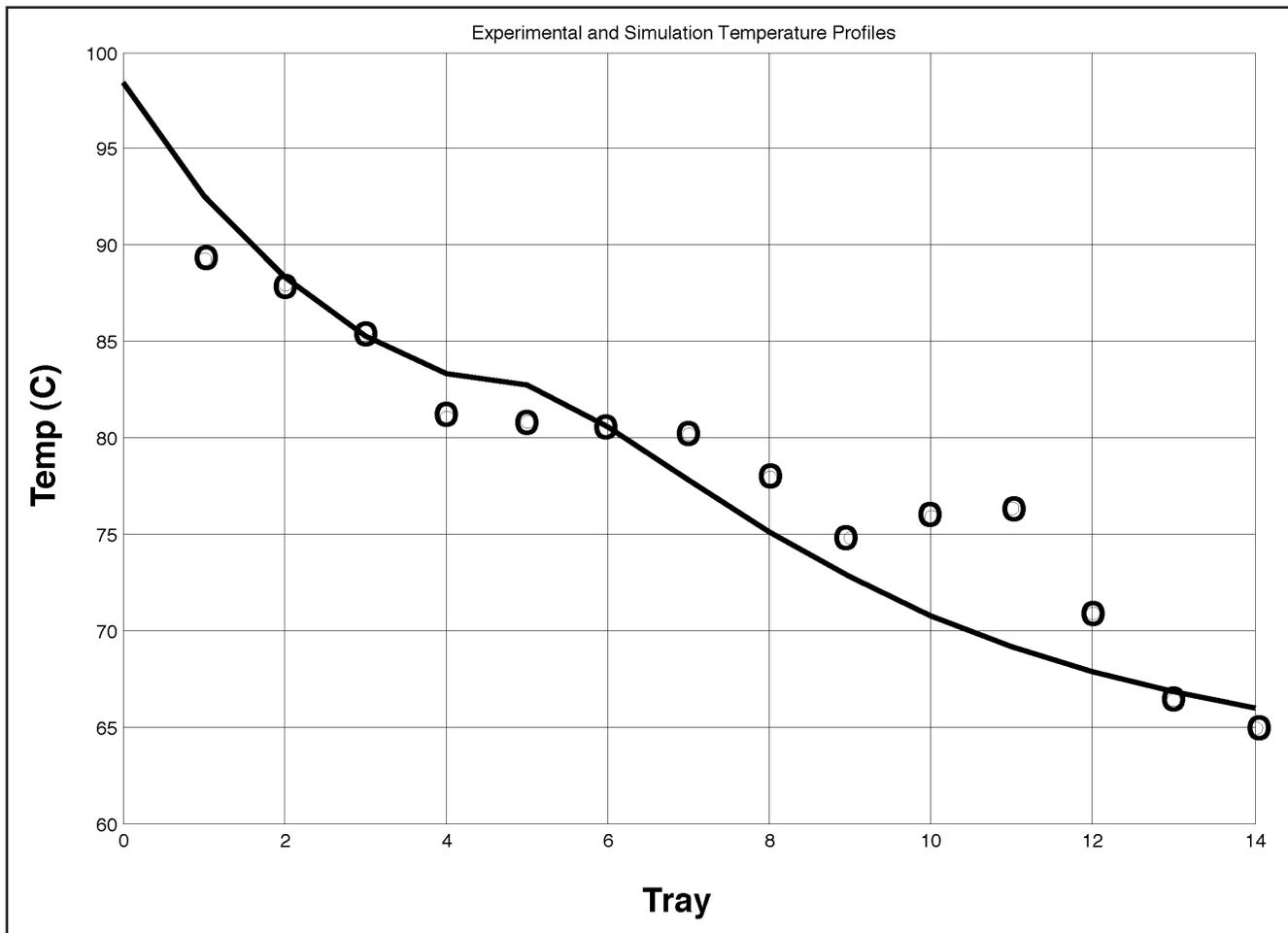


Figure 3. Temperature profiles; simulation and experimental diagram; total reflux.

The heat transferred into the feed in the preheater is:

$$\begin{aligned}
 Q_{PH} &= F_{adj} \cdot C_p \cdot (T_{out} - T_{in}) \\
 &= (1.631 \text{ kmol/h})(76.28 \text{ kJ/kmol-K})(62.6 - 20)(\text{h} / 3600 \text{ sec}) = 1.473 \text{ kW} \quad (4)
 \end{aligned}$$

Thus the total heat removed from the overhead vapor is 7.41 kW according to these cold-side calculations.

They can be checked by energy calculations on the hot process side. The heat removed in the three heat exchangers condenses and subcools the process vapor, which is the sum of the reflux and the distillate (0.464 + 0.345 = 0.809 kmol/h) with a composition of 0.954 mole fraction methanol. The enthalpy of the vapor leaving Tray 14 at 66.3 °C is 39,450 kJ/kmol, and the enthalpy of the subcooled liquid (reflux + distillate) is 37,900 kJ/kmol). Thus the total heat removed from the process vapor is:

$$\begin{aligned}
 Q_{total} &= (D+R)(H_{14} - h_R) \\
 &= (0.809 \text{ kmol/h})(39,450 - 3790 \text{ kJ/kmol})(\text{h} / 3600 \text{ sec}) = 8.104 \text{ kW} \quad (5)
 \end{aligned}$$

This independent check of the heat transferred in the overhead system is quite close to the 7.41 kW calculated from the cold sides of the preheater and condenser and is well within experimental error. There are some heat losses to the atmosphere from the hot vapor, so we would expect the hot-side heat removal to be higher than the cold-side heat pickup, which these calculation show to be the case.

The overall energy balance for the whole system shows a very significant heat loss. Most of the column shell is not insulated and at temperatures significantly higher than ambient. The reboiler duty is calculated from the

**The overall energy balance for the whole system shows a very significant heat loss.**

condensate flowrate and the difference between saturated steam at 35 psig and saturated condensate at 5 psig.

$$\begin{aligned} QR &= FS(H_{35} - h_5) \\ &= (0.433 \text{ kg/min})(2731 - 4546 \text{ kJ/kg})(\text{min}/600 \text{ sec}) = 16.4 \text{ kW} \end{aligned} \quad (6)$$

The overall energy balance is

$$\begin{aligned} \Delta H &= Q - W \\ D h_D + B h_B - F h_F &= QR - QCW - Q_{\text{loss}} \\ (0.345 \text{ kmol/h})(3790 \text{ kJ/kmol}) + (1.286 \text{ kmol/h})(7131 \text{ kJ/kmol}) \\ - (1.631 \text{ kmol/h})(1526 \text{ kJ/kmol}) &= 16.42 \text{ kW} - 5.94 \text{ kW} - Q_{\text{loss}} \\ 2.22 \text{ kW} &= 10.48 \text{ kW} - Q_{\text{loss}} \\ Q_{\text{loss}} &= 8.26 \text{ kW} \end{aligned} \quad (7)$$

Half of the reboiler duty is lost to the atmosphere.

### TOTAL REFLUX

Under total reflux conditions, there are no feed and no products. The liquid and vapor flowrates are the same in all sections of the column, which means the slope of the operating line is unity. Hence the operating line is the 45° line on the McCabe-Thiele diagram as shown in Figure 4.

*Under total reflux conditions, there are no feed and no products.*

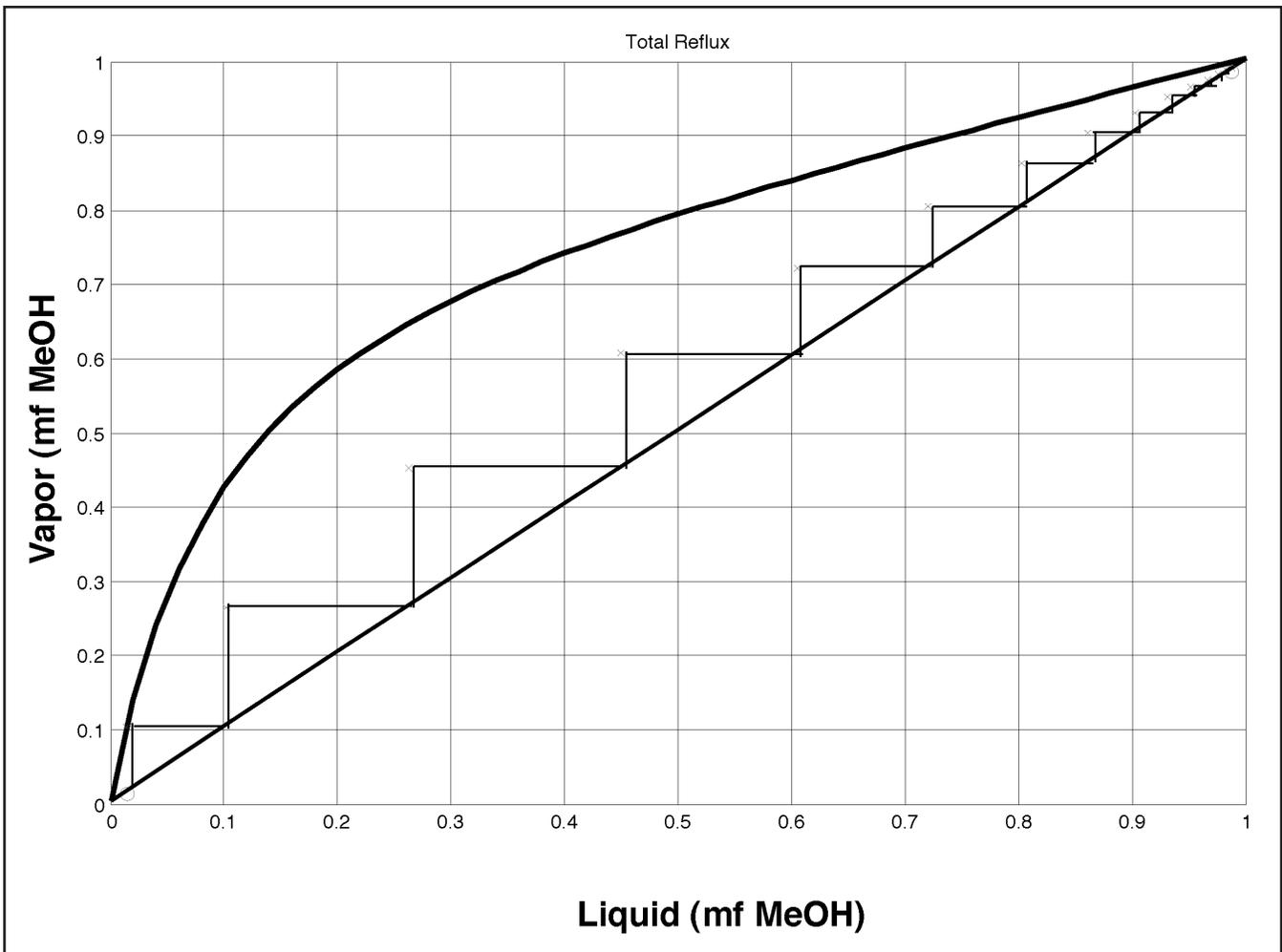


Figure 4. McCabe-Thiele.

Data from a total reflux run are given in Table 3. The specific gravity data are used to calculate the compositions of the reflux ( $x_R = 0.987$ ) and of the base ( $x_B = 0.0143$ ). Both of these purities are higher than in the feed run, as expected since the reflux ratio is infinite and more fractionation should occur.

The heat duty in the condenser from the cooling water data is 7.038 kW. From the overhead vapor, which is now only reflux, the duty is 8.368 kW. Heat losses could account for this difference.

The reboiler duty is calculated from the steam flowrate (13.54 kW). Since no streams are fed or leave the process, the  $\Delta H$  term in the overall energy balance is zero. Therefore the heat added in the reboiler should equal the heat lost in the condenser plus heat lost to the surroundings. The loss is  $13.54 - 7.038 = 6.50$  kW, which is similar to the heat loss in the feed run.

Figure 5 gives the experimental temperature profile (the circles) under total reflux conditions. The other curves come from the simulation and are discussed in the next section. Notice that the experimental temperature profile is fairly flat at both ends of the column, indicating quite high product purities.

## COMPUTER SIMULATION

An Aspen Plus simulation is developed to model the process. An Aspen "Radfrac stripper" model (reboiler but no condenser) is used for the column. Aspen "HeatX" models are used for the feed preheater and condensers. The NRTL physical property package is used. The process flow diagram is given in Figure 6.

	sp. gr.	Flowrate	Composition (mole fraction methanol)
Base	0.997	0	0.005
Reflux	0.793	559 cm <sup>3</sup> /min	0.995
Steam		0.357 kg/min	
CW		14.7 kg/min	
	Temperatures (°C)		
CW Into Condenser	6.0		
CW Out Condenser	12.9		
Reflux Drum	38.6		
Tray 1	99.4		
Tray 14	64.7		

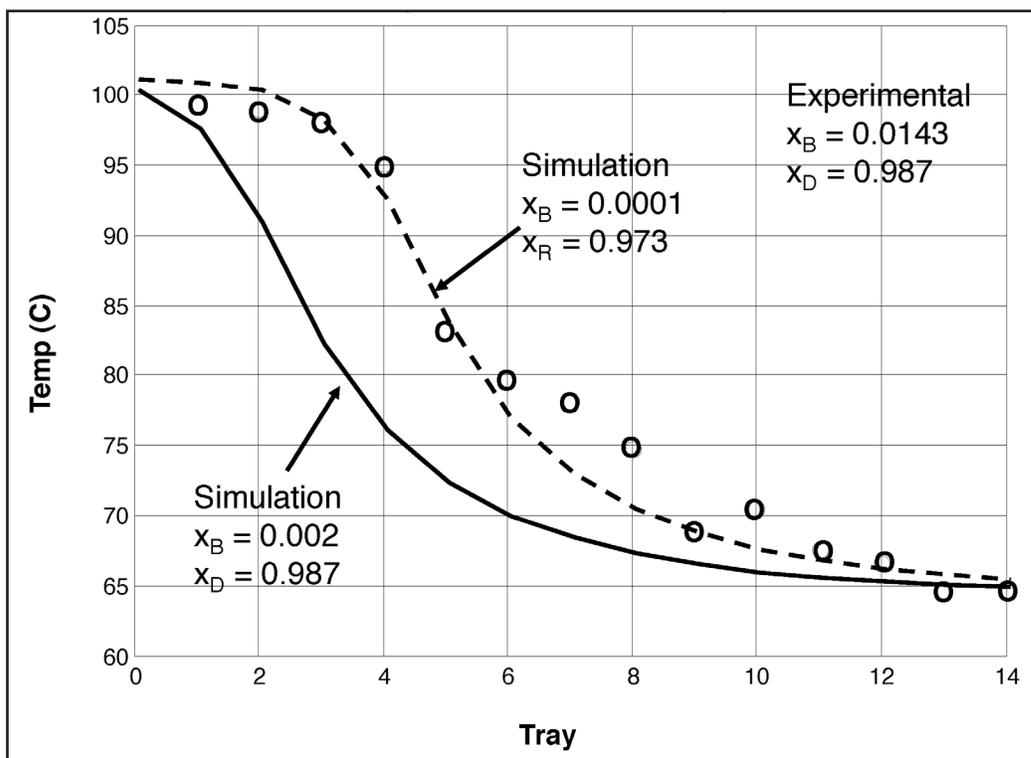


Figure 5. Temperature profiles; total reflux; simulation and experimental.

## Feed run

The experimental values that were specified for input data to the simulation are listed below.

1. Adjusted feed flowrate = 1.631 kmol/h

2. Adjusted feed composition = 0.2191 mole fraction methanol
3. Feed tank temperature = 20 °C
4. Bottoms flowrate = 1.286 kmol/h
5. Reflux flowrate = 0.464 kmol/h

6. Cooling water flowrate = 0.433 kg/min
7. Cooling water inlet temperature = 5.8 °C
8. Partial reboiler stage efficiency = 100%
9. Stage efficiency = 65%

All of the other variables are calculated by the model. Table 4 compares experimental and simulation results. The agreement is quite good. Figure 3 compares the temperature profiles.

		Experiment	Simulation
$x_D$	mf methanol	0.965	0.956
$x_B$	mf methanol	0.022	0.021
$Q_{PH}$	kW	1.473	1.547
$Q_{CW}$	kW	5.938	6.797
$Q_R$	kW	16.40	9.404
$T_{ovhd}$	°C	66.3	65.8
$T_{reflux}$	°C	50.6	46.9

### Total reflux run

How to set up a total reflux simulation is not completely obvious. The Aspen Plus “Radfrac” model requires feed and product streams, but these are all zero under total reflux operation. The reflux flowrate is set at the experimental value (0.8343 kmol/h). Efficiency is set at 65%.

A small feed flowrate (0.01 kmol/h) is specified with composition 0.211 mole fraction

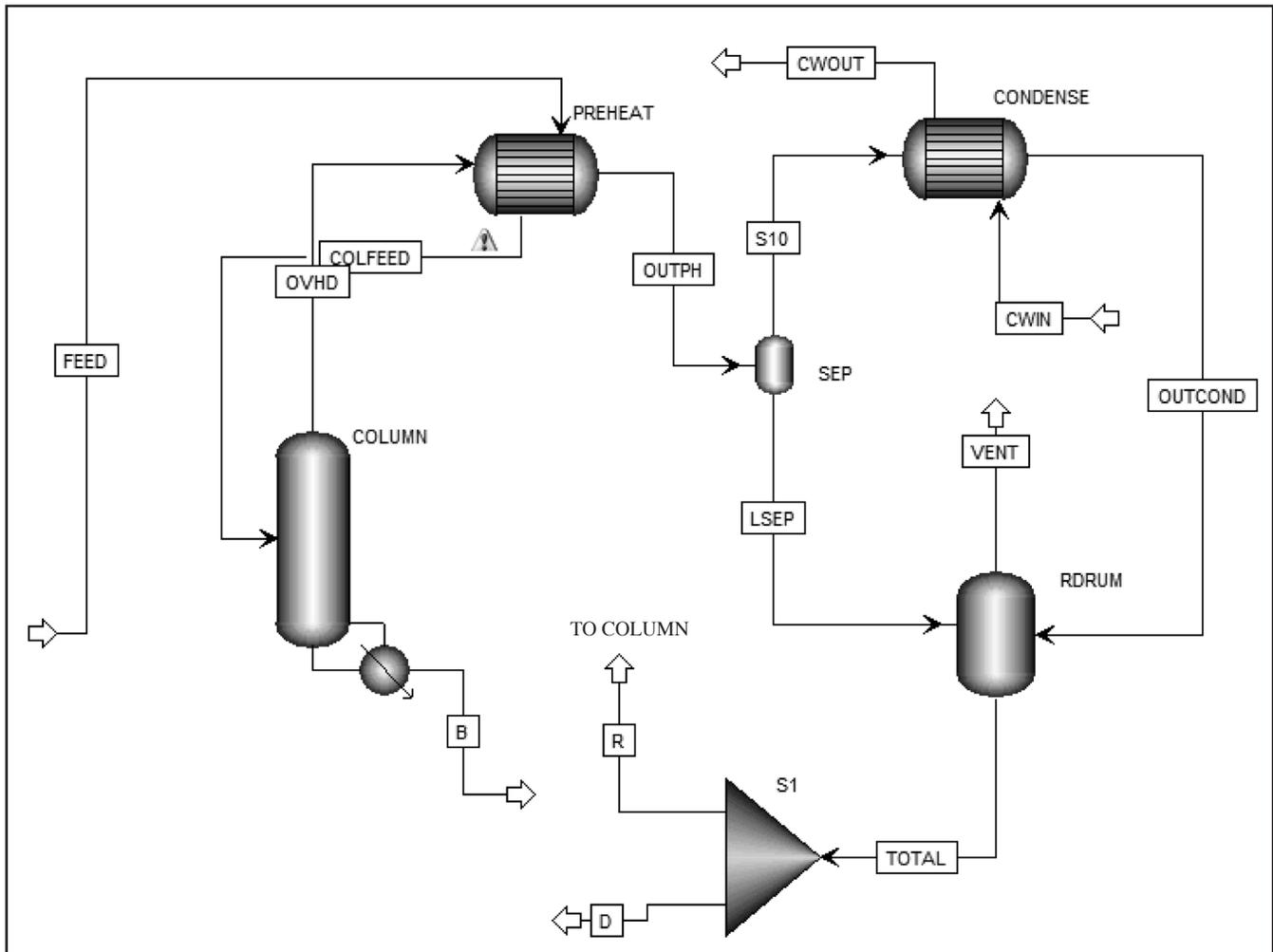


Figure 6. Aspen Plus process flow diagram.

methanol. Then the bottoms flowrate is varied and the calculated values of the reflux  $x_R$  and the base  $x_B$  are observed. A bottoms flowrate of 0.0078 kmol/h gives a reflux composition ( $x_R = 0.987$ ) that matches the experimental value. But the base composition is 0.002, which is much smaller than the experimental  $x_B = 0.0143$ . The predicted temperature profile under these conditions is the solid line shown in Figure 5.

The predicted temperature profile is not as flat in the lower part of the column as the experimental profile. This indicates that the real bottoms composition is lower than that calculated from density. Considerable inaccuracy is to be expected when purities are high.

A very small decrease in the specified bottoms flowrate to 0.00775 kmol/h changes the calculated compositions to  $x_R = 0.973$  and  $x_B = 0.0001$ . The resulting temperature profile is the dashed line in Figure 5.

There are multiple experimental temperature measurements and only one bottoms composition measurement, so it is logical to assume that the composition measurement is inaccurate.

## ASSESSMENT OF STUDENT LEARNING

Several student comments about the utility of the experiment are given below. The major contribution is permitting students to relate theory with real equipment.

1. *“The pilot-scale distillation experiment is very useful in understanding how a distillation column operates in real life. We were able to familiarize ourselves with proper start-up and shutdown procedures of the plant, and also how to make measurements of physical parameters such as the flow rate and the purities of the components. In addition, the energy requirements of the plant are also computed, which makes it very relevant to industrial operation.”*
2. *“I was honestly most excited to run this experiment because we had learned so much in theory and my design project was based upon distillation. It made it more real and helped with understanding how flow rates are actually measured and how the temperature rises up the column during start-up before reaching steady state. Those sorts of things aren't taught in the classroom.”*
3. *“The distillation experiment gave a lot of insight to how a distillation actually operates. We spent a lot of time in class studying how separations take place in distillation columns, and this lab allowed us to apply and actually*

*see the theories in action. The most interesting part about this experiment for me was seeing the difference between theory and actuality. Comparing the physical data to what is expected from theory and seeing how heat loss and efficiencies affect the results was a good learning experience.”*

4. *“In my opinion it was the most valuable experiment in the lab. The distillation experiment solidified the core foundations of mass transfer, as well as provided insight into proper unit operation beyond the textbook aspects. The experiment truly required teamwork. The construction of the unit allowed thorough understanding of the possible degrees of separation for a two-component system. The full-size column gave a unique experience in the senior operations lab.”*

## CONCLUSION

The experimental procedure and analysis of a realistically complex distillation experiment is described in detail. Educational objectives include reviewing mass transfer theory, developing a safe startup procedure, adjusting imperfect raw data, calculating tray efficiencies, and performing energy balances. Three generations of Lehigh chemical engineering students have found this experiment the most industrially relevant part of our two-semester senior-year process analysis laboratory. They gain very useful experience in operating a fairly complex process with multiple inputs and outputs that challenge their technical skill and develop their common sense.

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