



Distillation Technology and Modelling Techniques

Part 2: Shortcut distillation design methods

In this, the second part of their review, Konrad Miller and Katherine Shing demonstrate some shortcut distillation design methods such as the McCabe Thiele method and the Fenske Underwood Gilliland algorithm. Part 1 (February 2016) offered a conceptual framework of distillation and in the forthcoming final part three, Konrad Miller will apply these principles to the production processes of a modern brandy distillery.

Before the digital revolution, distillation columns, from petroleum fractionators to bourbon stills, were designed by hand calculation. Since coupled differential equations for heat transfer, mass transfer, and vapour-liquid equilibrium are extremely difficult and tedious to solve by hand, several brilliant 'shortcut' methods have been developed to rapidly analyse and size distillation columns.

Today, distillation columns are designed almost entirely via software simulation packages such as ASPEN, ChemCAD, and HYSIS. What possible

benefit is there to learning more archaic, shortcut methods? Why not just learn to use simulation software only? Of what benefit are these tools to the modern spirits distiller?

The answer is that an understanding of the shortcut methods is the quickest way to develop a physical intuition of the quantitative operation of a column. Many operational questions can be answered with this approach, such as understanding how a distillation will respond to a higher reflux ratio, a colder feed, or a variable stage count. Shortcut methods

provide graphical demonstrations and algebraic expressions that explain how column performance is dictated by these various constraints.

McCabe-Thiele

This shortcut method constructs 'operating lines', an expression of the material balance within the column, on an X-Y diagram. Figure 1 shows a ChemCAD generated X-Y diagram of the Ethanol-Water system at 1 atmosphere (14.7 psia) using the Non-Random Two Liquid (NRTL) model for solutions.

In this diagram, the x-axis is the mole fraction of ethanol in the liquid mixture, where $0 \leq x_{EtOH} \leq 1$. The y-axis is the mole fraction of ethanol in the vapour, also bounded from 0 to 1. The red line is the 'equilibrium curve', representing the vapour-liquid equilibrium. For example, a liquid with a 0.2 mole fraction ethanol is in equilibrium with a vapour of 0.54 mole fraction ethanol; while a liquid with an ethanol mole fraction of 0.6 is in equilibrium with a vapour of 0.7 mole fraction ethanol. The blue line simply serves as a guide for a 1:1 line.

There are several important things to note about this system: the vapour is consistently richer in

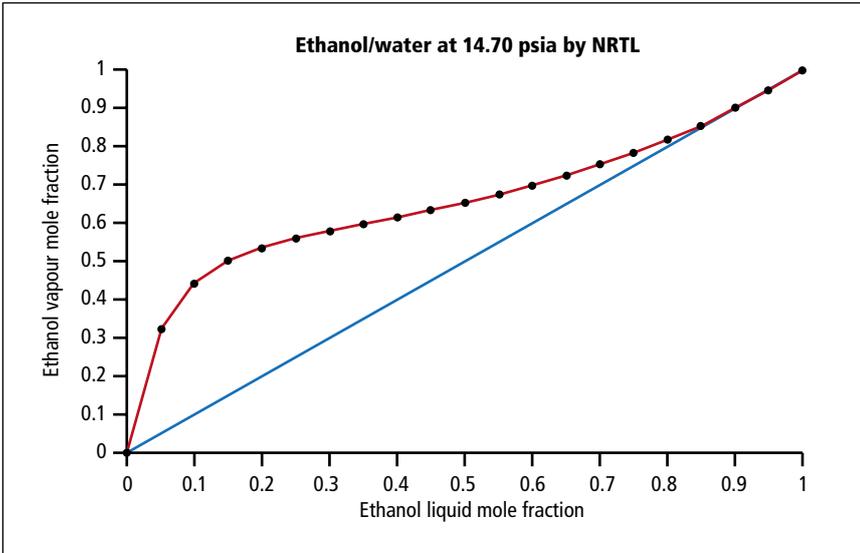


Figure 1: Ethanol-Water X-Y diagram

ethanol than the liquid which makes purification via distillation possible; and as the concentration of ethanol increases, the marginal increase from liquid to vapour decreases until around 0.9 mole fraction ethanol, where the liquid and vapour concentrations converge. This is known as an ‘azeotrope’ - a point in a solution where the liquid and vapour concentrations are identical.

Highly non-ideal solutions, such as ethanol-water, form azeotropes. This makes the production of 100% (also known as absolute) ethanol impossible by distillation at 1 atmosphere. Note that while pressure is fixed in the above diagram, temperature is not. The liquid/vapour solutions will be at 100°C (the boiling point of water) at the limiting case where the mole fraction ethanol is 0, and at 78.4°C (the boiling point of ethanol) at the limiting case where the mole fraction ethanol is 1.

We can now begin to construct the McCabe-Thiele diagram on the above X-Y plot. First, consider a mass balance between the top of the column, and an arbitrary tray ‘n’ some length down the column, where trays are numbered ‘1’ (top tray) down to ‘N’ (bottom tray). For this system, vapour enters from tray n+1 into tray n, liquid leaves from tray n down to tray n+1, and distillate leaves the system from the top of the column. Equation 1a gives the mass balance around this section and 1b gives the material balance around ethanol, the LK (Light Key) component, while Figure 2 highlights the area of interest. Note that ‘V’ refers to vapour flow, ‘L’ to liquid flow, ‘D’ to distillate, and ‘B’ to bottoms.

Eqn 1a:
 $V_{n+1} = L_n + D$

Eqn 1b:
 $y_{n+1} V_{n+1} = x_n L_n + \chi_D D$

We want to relate this mass balance to a correlation between x and y and superimpose it on the X-Y diagram. Solving for Y_{n+1} yields Equation 2:

Eqn 2:
 $y_{n+1} = \frac{L_n}{V_{n+1}} x_n + \frac{D}{V_{n+1}} x_D$

This equation relates the composition between the liquid and vapour of each stage to the liquid, vapour, distillate molar flow rates, and the compositions of each. If certain assumptions are made, such as constant molar overflow throughout the column, negligible enthalpic mixing effects, well insulated column, then Equa-

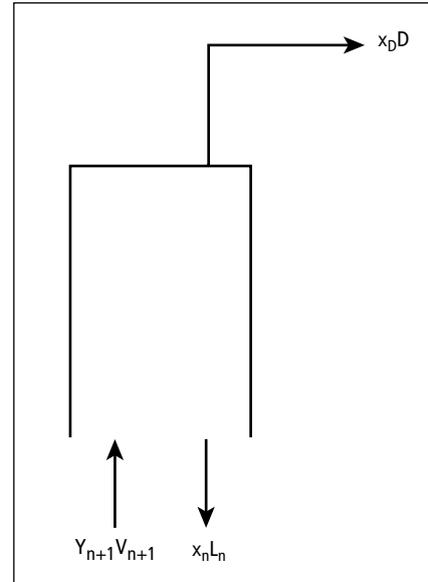


Figure 2: Material balance around the top part of a column

tion 2 can be extended throughout the top section of the column to produce Equation 3:

Eqn 3:
 $y = \frac{L}{V} x + \frac{D}{V} x_D$

Equation 3 is known as the ‘Operating Line’ for the ‘rectifying’ section. The rectifying section of a distillation column is generally above the feed and serves to purify the LK component in the distillate. Note that L/V cannot be greater than 1 as this corresponds to ‘total reflux’, where all the condensed vapour is return back to the column rather than drawn away as distillate (recall reflux is liquid returned from the condenser to the column). We now see why increasing the amount of liquid refluxed increases the purity of

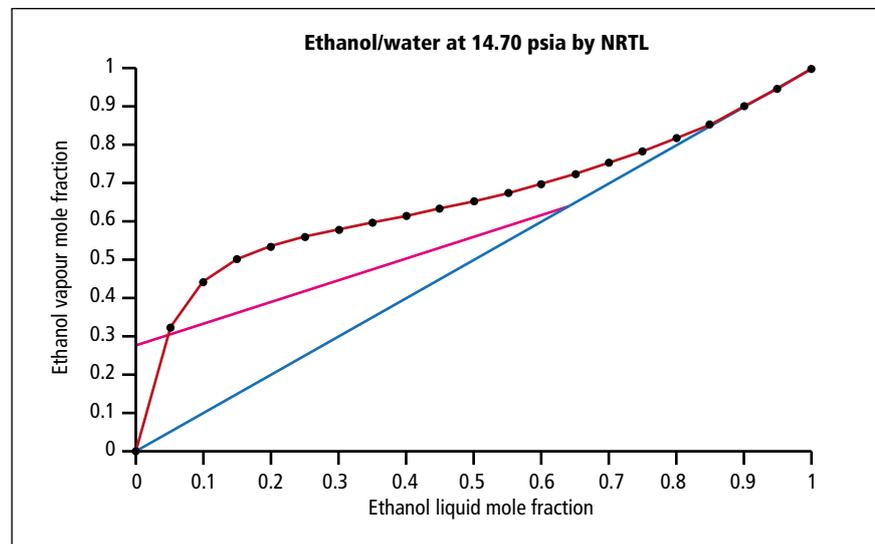


Figure 3: Rectifying Line superimposed on X-Y diagram. Note that the line intersects the ‘45 Degree Line’ where x_D is specified. The slope is given by $(\frac{RR}{RR+1})$, and the y intercept is $(\frac{1}{RR+1})x_D$

the LK in the distillate: L/V increases, increasing 'y', the concentration of ethanol leaving the top of the column.

There is one last useful modification to the Rectifying Line equation: Let the 'Reflux Ratio' (RR) be defined as ratio of liquid reflux, L, returned to the column from the condenser to the amount of liquid distillate, D, drawn away from the condenser as product, or $RR = L/D$. At the limiting case of $RR = L/D \rightarrow \infty$, all material going to the condenser is returned as reflux (Total Reflux), and $L/V=1$. The opposite case is where no reflux is returned: $RR = L/D \rightarrow 0$, $L/V = 0$. If we substitute this into Equation 3, we can specify the entire rectifying section operation by RR and x_D , as seen in Equation 4:

Eqn 4:

$$y = \left(\frac{RR}{RR+1}\right)x + \left(\frac{1}{RR+1}\right)x_D$$

With this in mind, we can superimpose the Rectifying Line over the X-Y diagram, with a distillate of 0.65 mole fraction ethanol and a reflux ratio of 1.3. Figure 3 shows the results.

We can now apply the same analysis to the area below the column, or the "Stripping Section". The Stripping section of a distillation column is generally below the feed, and serves to purify the HK component in the bottoms. If we define the 'Boilup Ratio' (BR), to be equal the ratio of vapour driven up the column (boilup, V) and the rate of liquid product drawn from the column (Bottoms, B), then we can derive the Operating line for the Stripping Section, Equation 5:

Eqn 5:

$$y = \left(\frac{BR+1}{BR}\right)x - \left(\frac{1}{BR}\right)x_B$$

From this, we can see how increasing the Boilup Ratio decreases the concentration of the LK in the bottoms, thus increasing the purity of the HK in the bottoms. This too should be superimposed over the X-Y diagram. Figure 4 shows the Stripping Line, with a bottoms product of 5% Ethanol and a Boilup Ratio of 0.25.

We have now taken into consideration the distillate composition, the Reflux Ratio, the bottoms composition, and the Boilup Ratio. To finish specifying our column, we must consider the feed composition and phase. The final operating curve is known as the 'Q-Line', and takes into account both

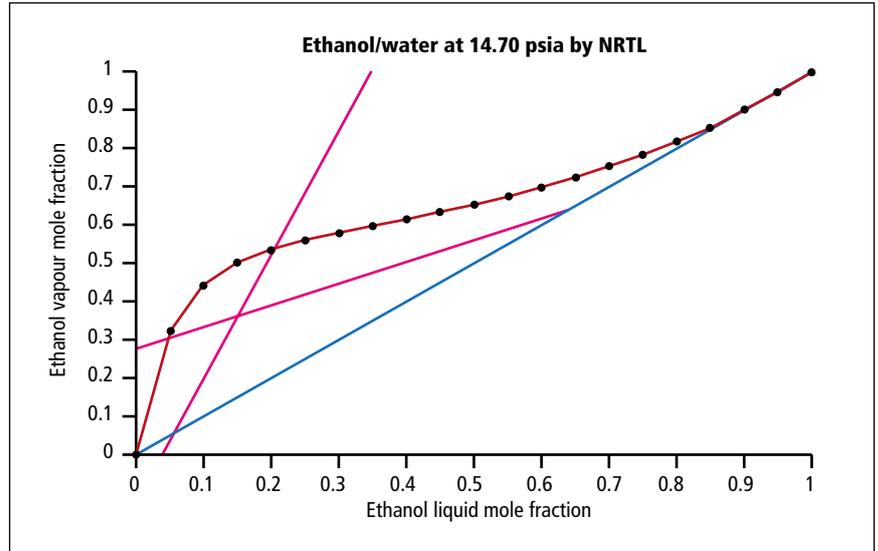


Figure 4: Stripping and Rectifying Lines superimposed on X-Y diagram

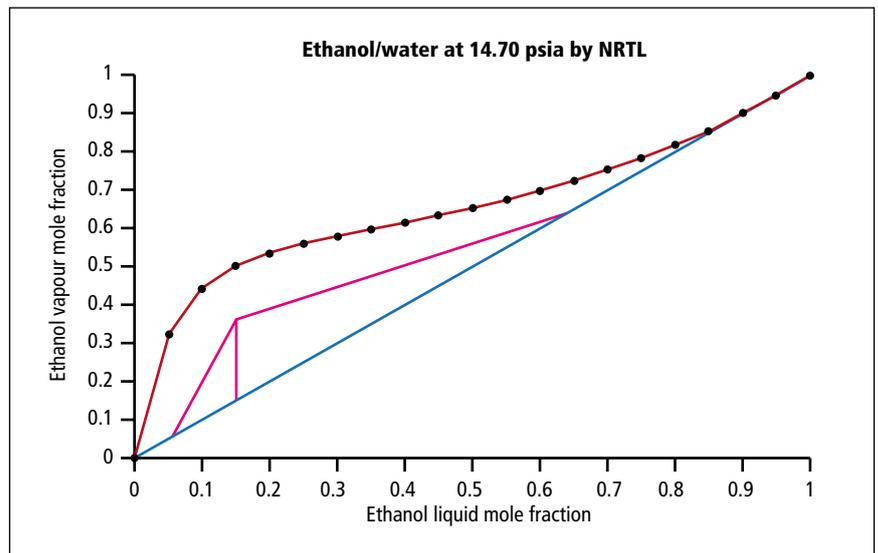


Figure 5: Rectifying, Stripping, and Q-Lines on an X-Y diagram

the composition and phase of the feed. The q parameter used in the equation is a measure of the amount of heat needed to fully vaporize the feed, and is defined in Equation 6:

Eqn 6:

$$q = \frac{\text{Heat to make feed a saturated vapor}}{\text{Heat of Vaporization of feed}}$$

For a liquid at the boiling point, $q = 1$. Since most industrial ethanol distillations take place with a near-saturated liquid feed, we will assume $q=1$. The Q-Line equation is given in Equation 7:

Eqn 7:

$$y = \left(\frac{q}{q-1}\right)x - \left(\frac{1}{q-1}\right)z_F$$

Recall that all x , y , and z variables are given in terms of the LK (ethanol). We can plot the Q-Line for $q=1$ and a feed $z_F = 0.15$ (similar to wine). Figure 5

shows the Rectifying Line, Stripping Line, and Q-Line on an X-Y diagram:

Note how all three lines converge at one point. We have selected our column variables such that this is the case. A properly constructed McCabe-Thiele diagram does not need to exhibit this, but a designer should always strive for convergence when specifying a separation.

Once the operating conditions for the feeds and draws from the column have been defined, we can determine the number of stages required. We do this by 'stepping off' stages along the McCabe-Thiele diagram. Starting at one intersection of an Operating Line (either the Rectifying Line or the Stripping Line) and the 45 degree line, we build a 'Staircase' between the Operating Lines and the Equilibrium Line. Each 'stair' represents a stage, and describes the liquid and vapour composition at each stage along

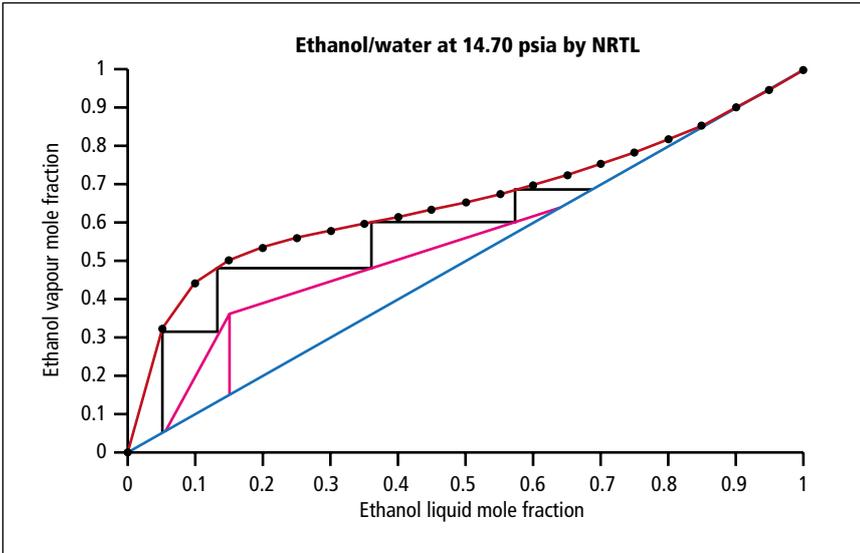


Figure 6: McCabe-Thiele diagram for Ethanol-Water separation

the column. Figure 6 shows a fully completed McCabe-Thiele diagram for an ethanol-water separation, with a saturated liquid feed at 15% ethanol (i.e. wine), a reflux ratio of 1.3, a distillate of 65% ethanol, a Boilup Ratio of 0.25, and a bottoms product of 5% ethanol:

In this instance, four stages are required. Stages should be counted from top to bottom, with the very top as stage 1 and the very bottom stage 4. If a partial (as opposed to a total) condenser is used, then the

Stage	X_EtOH	Y_EtOH
1	0.57	0.69
2	0.36	0.6
3	0.14	0.48
4	0.05	0.32

Table 2: Vapour and liquid composition along the column

condenser serves as an additional equilibrium stage; if a reboiler is used (as opposed to steam injection), then the reboiler is the bottom stage. Finally, the feed stage should be specified where the Operating Lines intersect. Here, stage 3 is the optimal feed point.

We can read the composition on each tray from the McCabe-Thiele Diagram as well, by looking at where the stages touch the Equilibrium Curve. Table 1 gives the compositions along the column:

A few things of note: first, the vapour leaving stage 1 is condensed, and a fraction of it is removed as distillate (the remaining is returned as reflux). The same holds true for the liquid in stage 4 for the reboiler. Second, the staircase construction was started on this diagram at the bottom of the Stripping Line. Hence the

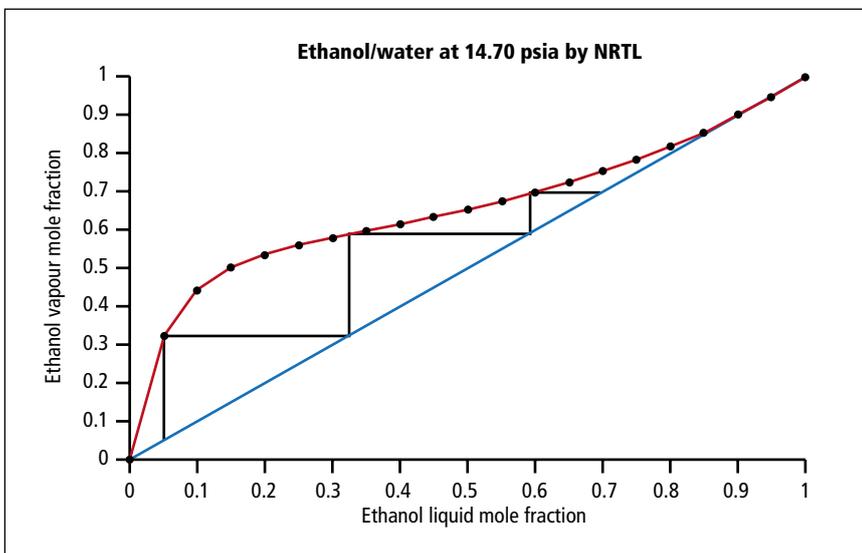


Figure 7: McCabe Thiele diagram at Total Reflux, minimum number of stages. Minimum 3 stages required for the specified split at total reflux

bottoms composition is 5% ethanol, but the distillate is 69%, rather than the specified 65%, due to staircase construction. Had we started at the top, the distillate would have been 65%, but the bottoms <4%. It is very rare that the number of stepped off stages equates exactly to the desired split, and an additional stage is usually required to achieve the minimum required purity. Industrial ethanol separations typically operate with much more stringent requirements: higher product purities, near zero loss of ethanol through the bottoms, and low boilup and reflux rates (to minimise operating costs) dictate columns in the 50-100 stage range.

The McCabe-Thiele method calculates the number of equilibrium stages required for a specific separation. Actual trays rarely reach perfect equilibrium, so some overall column efficiency is often assumed. Bubble cap trays usually offer around ~80% efficiency, valve trays offer ~70% efficiency, and sieve trays usually offer ~55% efficiency (these are gross estimates, actual efficiencies will vary wildly according the specific system and tray).

In beverage ethanol distillations, bubble cap trays are typically used in the rectifying section for their high efficiency and turndown, while sieve trays are used in the stripping section for their ability to handle any solids in the feed. In this example, 3 (2.5 calculated) bubble cap trays and 4 (3.6 calculated) sieve trays would be specified for this column.

When all material entering the condenser and reboiler is returned into the column as reflux or boilup, a scenario known as 'Total Reflux' is achieved. The operating lines reach their peak efficiency and converge onto the 45 degree line. The minimum number of stages for a given split can then be stepped off in the typical McCabe Thiele manner. Figure 7 illustrates this example:

The other extreme is minimum reflux. In this scenario, the Reflux and Boilup Ratios are decreased until either the intersection of the Operating Lines touch the Equilibrium Curve, or one of the Operating Lines becomes tangent to the equilibrium curve. This point of contact is known as a 'Pinch Point' - an infinite number of stages are required, as the Operating Lines or Line converges to equilibrium. Figure 8 shows the McCabe-Thiele construction for minimum reflux, infinite stages:

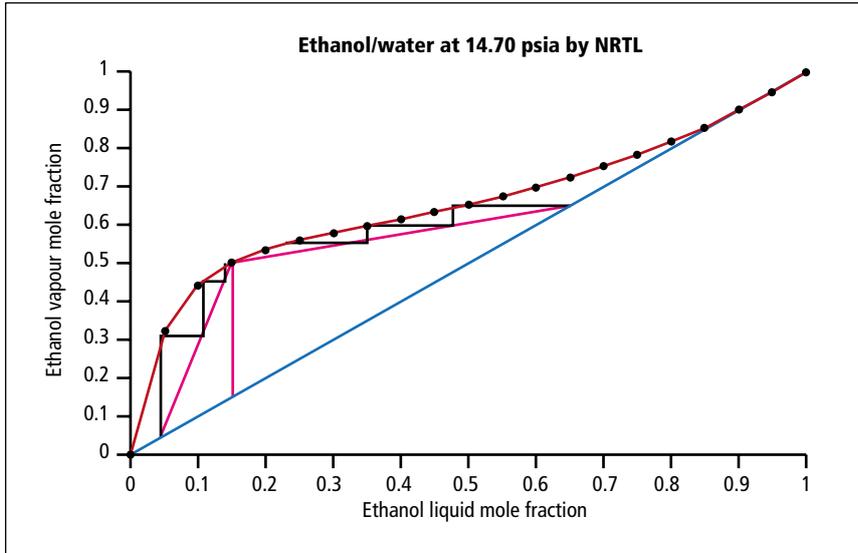


Figure 8: McCabe-Thiele diagram for Minimum Reflux, infinite stages

The calculated Minimum Reflux for this split is 0.44, and the Minimum Boilup is 0.22. Note how the stages get smaller and smaller (indicating less separation) as they approach the Pinch Point. McCabe-Thiele plots are also extremely useful in the field for locating Pinch Zones in already existing columns.

Fenske-Underwood-Gilliland method

This technique is used to specify the approximate number of stages and the approximate reflux ratio for a multi-component distillation. As mentioned previously, multicomponent distillation analysis is enormously simplified when considered as a pseudo-binary split between the Light Key and Heavy Key components.

Like the McCabe-Thiele technique, the Fenske-Underwood-Gilliland (FUG) method has largely been replaced by more rigorous computer modelling.

However, many simulation packages still offer the FUG method due to its swift convergence for approximate models and utility as a starting point for iterative solvers.

The first step in the FUG method is to apply the Fenske Equation to determine the minimum number of stages for a give separation. To solve this, the mole fraction of the LK in the distillate and bottoms, and the mole fraction of the HK in the distillate and bottoms must be specified. Finally, the relative volatility between the two Key components, $\alpha_{LK, HK}$, must be determined. If the relative volatility is not constant throughout the length of the column, the geometric mean can be employed instead. Note that the Fenske equation is also applicable to binary distillation, where $x_{LK} + x_{HK} = 1$. Once the split between the LK and HK is defined, Equation 8 can be used to solve for N_{min} .

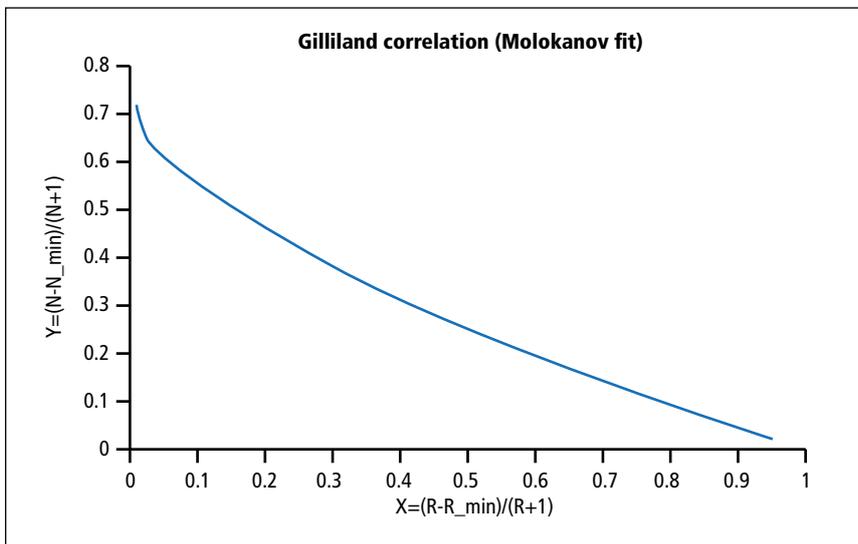


Figure 9: Molokanov's fit to the Gilliland Correlation for reflux and stage count

Eqn 8:

$$N_{min} = \frac{\log \left[\frac{x_{LK,D} \cdot x_{HK,B}}{x_{LK,B} \cdot x_{HK,D}} \right]}{\log (\alpha_{LK, HK})}$$

Next, the Underwood equation can be used to determine the minimum reflux required for a given separation. The Underwood equation is divided into two 'classes' of application. Class 1 separations (Equation 9a) result when every component in the feed distributes in some quantity to both the bottoms and the distillate. Class 2 separations (Equations 9b and 9c) occur when one or more feed component does not partition to the bottoms or the distillate. In both cases, the distribution of any two components must be specified. Since splits are usually defined between the LK and HK, i and j are often set to LK and HK in Equations 9a:

Eqn 9a (Class 1):

$$R_{min} = \frac{(x_{i,D}/z_{i,F}) - \alpha_{i,j} (x_{i,D}/z_{j,F})}{\alpha_{i,j} - 1}$$

Eqn 9b (Class 2):

$$\sum \text{species } i \frac{\alpha_{i, HK} x_{i,D}}{\alpha_{i, HK} - \Phi} = 1 + R_{min} \text{ where}$$

Eqn 9c (Class 2):

$$\sum \text{species } i \frac{\alpha_{i, HK} z_{i,F}}{\alpha_{i, HK} - \Phi} = 1 - q$$

In the event of a Class 1 separation, Eqn 9 can be evaluated directly. In the event of a Class 2 separation, Eqn 9 can be solved for the Φ parameter, and then Equation 9b can be solved.

Equations 8 and 9 provide the means for determining the minimum stages and the minimum Reflux Ratio required for a separation. These ideal values are not practical to use, we now need to find 'actual' stage count and reflux ratio. We find these real values from the minimum values by using the Gilliland correlation.

Gilliland correlation

The Gilliland correlation is the result of Robinson's and Gilliland's research in correlating and to the actual number of stages and Reflux Ratio. Gilliland's correlation is well described by Molokanov's expression (Equation 10):

Eqn 10:

$$Y = \frac{N - N_{min}}{N + 1} = 1 - \exp \left\{ \left(\frac{1 + 54.4X}{11 + 117.2X} \right) \left(\frac{X - 1}{\sqrt{X}} \right) \right\},$$

where $X = \frac{R - R_{min}}{R + 1}$

The resulting curve is shown in Figure 9:

With the actual number of stages and actual reflux ratio determined, the last step is to determine the feed stage. While the Fenske Equation can be turned 'inside-out' to calculate the feed stage, the result typically varies greatly from reality. Kirkbridge developed the following relationship (Equation 11) between the number of rectifying stages, N_R , the number of stripping stages, N_S , and the bottoms (B) and distillate (D) flow rates:

Eqn 11:

$$\frac{N_R}{N_S} = \left[\left(\frac{z_{HK,F}}{z_{LK,F}} \right) \left(\frac{z_{HK,F}}{z_{LK,F}} \right)^2 \frac{B}{D} \right]^{0.206}$$

We now have the tools to rapidly estimate the number of stages, the reflux ratio, and the feed point for a multicomponent column. For either the McCabe-Thiele case or the FUG case, reboiler and condenser duties can be estimated by determining the reflux and boilup flows, finding the enthalpy of vaporisation of the distillate and bottoms, and performing an enthalpy balance on the entire column. Part

three of this feature will build on this foundation to examine the operation of a distillery employing continuous distillation.

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Konrad Miller is a process engineer from the United States. A licensed professional engineer, he graduated from the University of California, Berkeley with a BS in chemical engineering and from the University of Southern California with an MS in chemical engineering. His work as a process engineer in the alcoholic beverage industry has included process design, plant simulation, fermentation, and distillation.

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