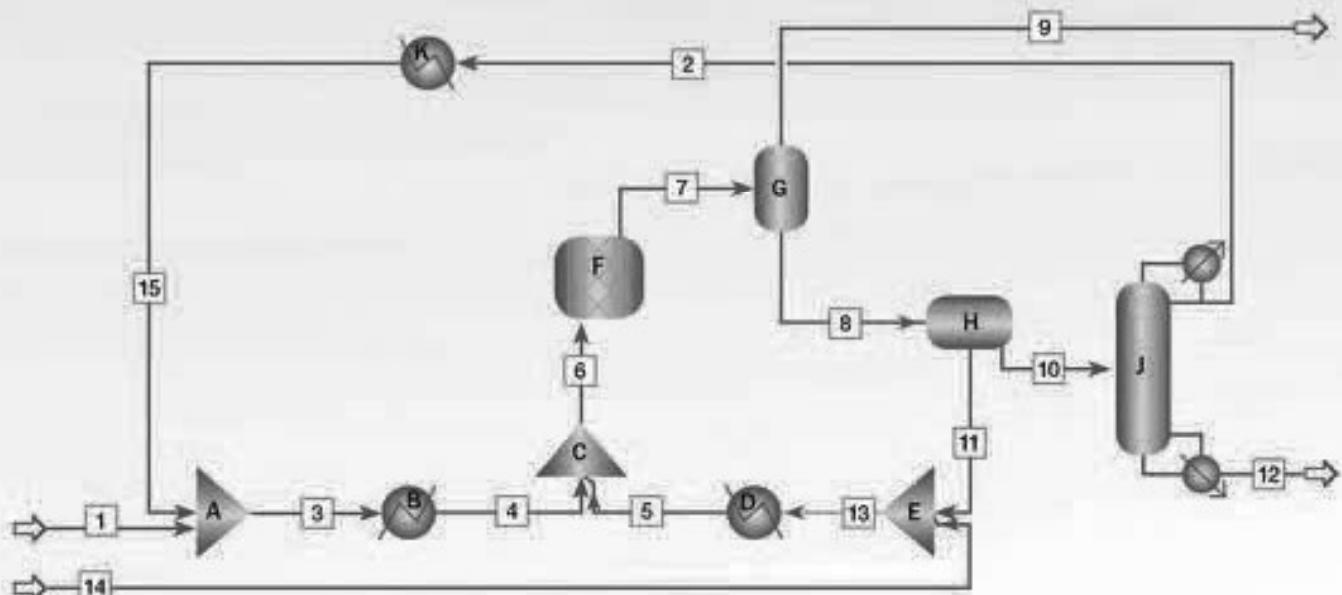


Ralph Schefflan

# Teach Yourself the Basics of Aspen Plus



Second Edition



AIChE

WILEY

# Chapter Eleven

## Multistage Equilibrium Separators

### 11.1 Introduction

Distillation and extraction are the most commonly used separation methods in chemical processing, and models of these are the heart of Aspen Plus. All of the Aspen Plus distillation and extraction models can be found in the model library under the Columns tab.

For a distillation column, simulation implies the existence of a fully configured column, that is, the number of theoretical stages and the feed(s) and side stream(s) locations. When the state of the feeds and the overhead, bottoms, and side stream specifications are provided, the model calculates the state of the products and the reboiler and condenser heat loads if they were not explicitly specified. For an extraction column, the same ideas apply, except that there are no overhead and bottoms specifications required. There is limited capability in Aspen Plus to directly design a column to yield products of specified composition, thus the technique is to search the solution space for a trial column with the desired characteristics, and, therefore, many rating solutions may be required until one that matches the desired design specifications is obtained.

### 11.2 The Basic Equations

All models of distillation and extraction processes involve the solution of the material, equilibrium, and energy balance equations or an applicable subset. All models are based on the analysis of a single stage, such as shown in [Figure 11.1](#). There are several ways to write the describing equations, based on the choice of independent variables. For the set of equations given here, the choices  $v_i^j$  and  $l_i^j$ , the component  $i$ , vapor and liquid leaving stage  $j$ ,  $f_i^j$ , the componential feed to stage  $j$ ,  $s_i^j$ , a componential side stream leaving stage  $j$ ,  $T_j$  and  $P_j$ , are the temperature and pressure of stage  $j$ , and  $Q^j$  is the total energy entering or leaving stage  $j$  are used. The phase equilibrium constant  $K_i^j$  depends on the stage temperature, pressure, and liquid and vapor mole fractions,  $x_i^j$  and  $y_i^j$ . Mole fractions can be expressed in terms of componential molar flows, for example, a liquid mole fraction can be calculated by [Equation 11.1](#), where  $m$  is the number of components.

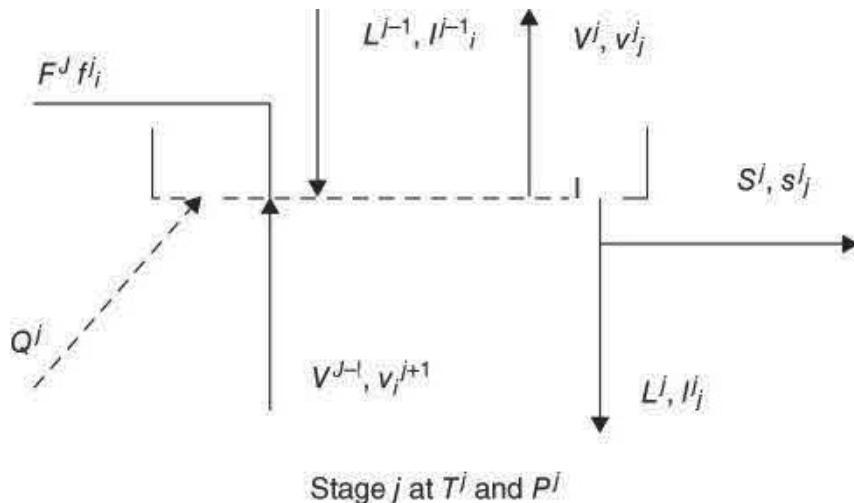
$$x_i^j = \frac{l_i^j}{\sum_{m=1,n} l_m^j} \quad \text{11.1}$$

$V^j$  and  $L^j$  are auxiliary variables that refer to the total molar vapor and liquid leaving stage  $j$ , and are calculated by summing the componential flows in the vapor and liquid leaving stage as shown in [Equation 11.2](#).

$$V_j = \sum_{m=1,n} v_m^j \quad 11.2$$

$$L_j = \sum_{m=1,n} l_m^j$$

$H^j$  and  $h^j$  are the molar enthalpies of the vapor and liquid leaving stage  $j$ , which depend on composition, temperature, and pressure.



**Figure 11.1** A theoretical stage.

The material balance for component  $i$ , on stage  $j$ , is given in [Equation 11.3](#). Here,  $s_i^j$  refers to the molar flow of component  $i$  in a side stream, if it exists.

$$f_i^j + v_i^{j-1} + l_i^{j+1} - v_i^j - l_i^j - s_i^j = 0 \quad 11.3$$

There are  $nm$  material balances, where  $n$  is the number of theoretical stages and  $m$  is the number of components. The introduction of a side stream adds one degree of freedom since the state of a side stream is equal to the state of its source, that is, the phase of the stage from which it is removed, but its flow rate is unknown; however, the composition, temperature, and pressure of the side stream are known. A specification for a liquid side stream, such as the ratio of the side stream to the liquid, removes the extra degree of freedom and permits the added calculation of the componential flows of the side stream.

The basic equilibrium equations for component  $i$ , on stage  $j$ , are given in [Equation 11.4](#).

$$y_i^j = K_i^j x_i^j \quad 11.4$$

When the independent variables and [Equation 11.2](#) are substituted into [Equation 11.4](#), [Equation 11.5](#) is obtained. There are  $nm$  equilibrium equations.

$$v_i^j = K_i^j(v_i^j, l_i^j, T^j, P^j) \frac{\sum_m^j v_m^j}{\sum_m l_m^j}$$

The enthalpy per mole of liquid and vapor flows  $H^j$  and  $h^j$  refer to the vapor and liquid leaving stage  $j$ , respectively. For each stage, an energy balance, given by [Equation 11.6](#), may be written, where  $S^j$  represents the total molar flow of a side stream. The enthalpy/mole of a side stream depends on its source and may be written as either  $H^j$  or  $h^j$  depending on the state of the side stream.

$$H_F^j \sum_m f_m^j + H^{j-1} \sum_m v_m^{j-1} + h^{j+1} \sum_m l_m^{j+1} - H^j \sum_m v_m^j - h^j \sum_m l_m^j - h^j \sum_m S_m^j + Q^j = 0 \quad 11.6$$

If there are no feeds, side streams, or heat exchangers associated with stage  $j$ , the  $F$ ,  $S$ , and  $Q$  terms disappear. There are  $n$  energy balance equations.

Models usually do not include stage pressure calculations and pressures are normally assigned a priori. The summation of equations for a distillation column is as follows:

material balances	$nm$
equilibrium equations	$nm$
energy balances	$m$
Total	$m(2n + 1)$ .

In the absence of side streams or interstage heat exchangers, a count of unknowns for a column with a condenser and a reboiler is as follows:

componential vapor flows	$nm$
componential liquid flows	$nm$
stage temperatures	$m$
condenser and reboiler duties	2
Total	$m(2n + 1) + 2$

Thus, there are typically two degrees of freedom that correspond to the column's end specifications. These may be reboiler and condenser duty specifications. Other specifications that can be used such as product rate and reflux ratio require minor modifications to the basic equations.

Each side stream adds one degree of freedom; therefore, it is necessary to provide two end specifications, plus a specification such as a side stream flow rate, for each side stream.

For extraction columns, the equations are identical to those of distillation columns if one recognizes that the vapor terminology now refers to the lighter

of the two phases. Additionally, since there are no reboilers or condensers, the degrees of freedom are zero except that one additional degree of freedom is introduced for each side stream as in distillation columns.

The set of equations described above lend themselves to solution simultaneously by means of the Newton–Raphson method, such as the algorithm of Naphtali and Sandholm (1971). This method has the advantage of permitting the use of all possible end and side stream specifications; however, it has the disadvantage of requiring starting estimates for all independent variables.

There are many arrangements of the equations with an accompanying convenient selection of variables that lead to a variety of algorithms to generate a solution. For example, one could employ total flows and mole fractions. There are a group of algorithms that alternately solve the locally linear material balances and then the nonlinear energy balances adjusting the  $K$  values between iterations. The method used in Aspen Plus's RadFrac block is the inside–out algorithm of Boston and Britt (1974), but the implementation is proprietary. Details of many methods can be found in Seader and Henley (1998).

### 11.3 The Design Problem

In order to simulate a distillation column within a process, it is necessary to initially solve a design problem, that is, for a given service, to establish the number of stages required, the location of the feeds and side streams, and define suitable end specifications. Since the rigorous distillation models in Aspen Plus do not directly calculate design parameters, such as the number of stages and the feed location, it is necessary to explore many possible rating solutions. Prior to the use of a rigorous model, it is useful to estimate the design by less rigorous methods including the manual McCabe–Thiele method (1925). Prior to any design work, it is imperative that the vapor–liquid equilibrium diagrams and/or liquid–liquid equilibrium diagrams be reviewed. In many cases, the data stored in the Aspen Plus database will be suitable, but this may not necessarily be the case and a literature search or experimental work may be required.

A brief review of the McCabe–Thiele method is given in what follows. Unlike the models in Aspen Plus, the McCabe–Thiele method can be used to solve the design problem, that is, given the feed composition, flow rate, thermal state, and product composition targets, determine feed location and the number of stages above and below the feed stage, but is only applicable to two-component systems except in a special way to extractive distillation. The following features are the heart of the method.

- 1.** All stages are at steady state and the stage products are at equilibrium.
- 2.** The column operates under equal molal overflow conditions, that is, within a section of a distillation column where there is no feed, no product

takeoff, and no interstage heat exchangers, the flow rates of all liquids leaving a stage are identical as are the flow rates of all vapors.

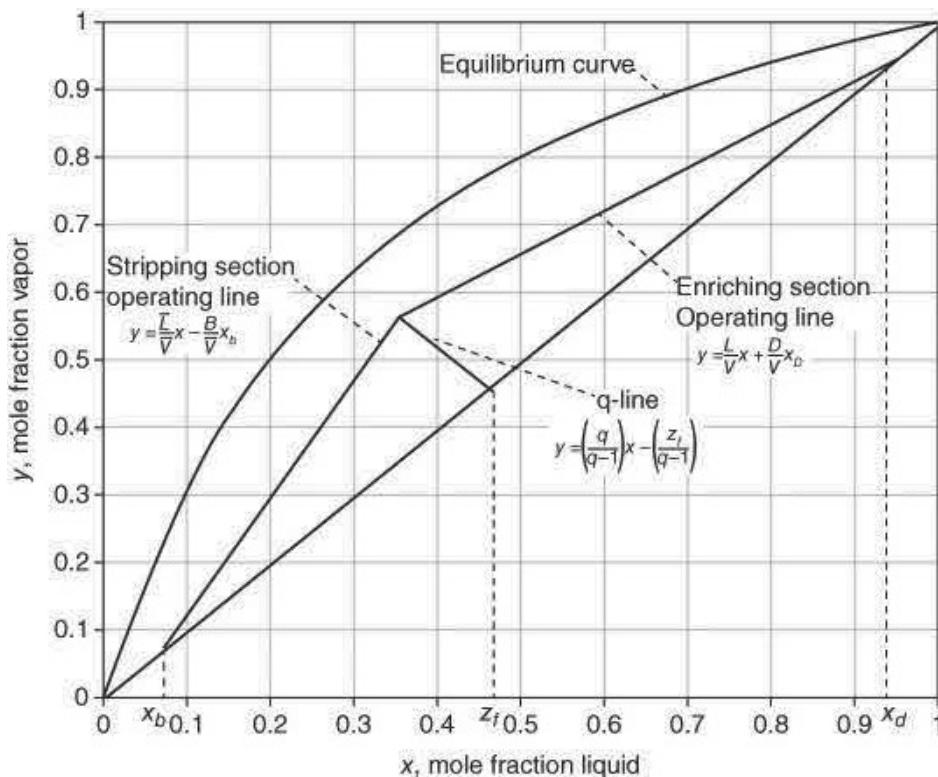
**3.** After a solution has been obtained, the reboiler and condenser duties are calculated by a manual energy balance around each device.

The McCabe–Thiele method can also be used to rate columns by a trial-and-error procedure.

An overview of the method is given with reference to [Figure 11.2](#), the McCabe–Thiele diagram. The coordinates are the vapor and liquid mole fractions. An equilibrium curve based on experimental data is plotted on the figure. There are operating lines above and below the feed that are derived from the material balance equations in terms of the vapor and liquid mole fractions. The equations of the two operating lines are given in the figure. The two operating lines intersect with the  $y = x$  line at the column's respective end compositions and intersect with a feed line that emanates from the feed composition,  $z_f$ , located on the  $y = x$  line. The slope of the feed line,  $q$ , is calculated by [Equation 11.7](#), where  $H_F$  represents the molar enthalpy of the feed,  $H_f$  is the molar enthalpy of saturated vapor, and  $h_f$  is the molar enthalpy of saturated liquid.

$$q = \frac{H_f - H_F}{H_f - h_f} \quad \text{11.7}$$

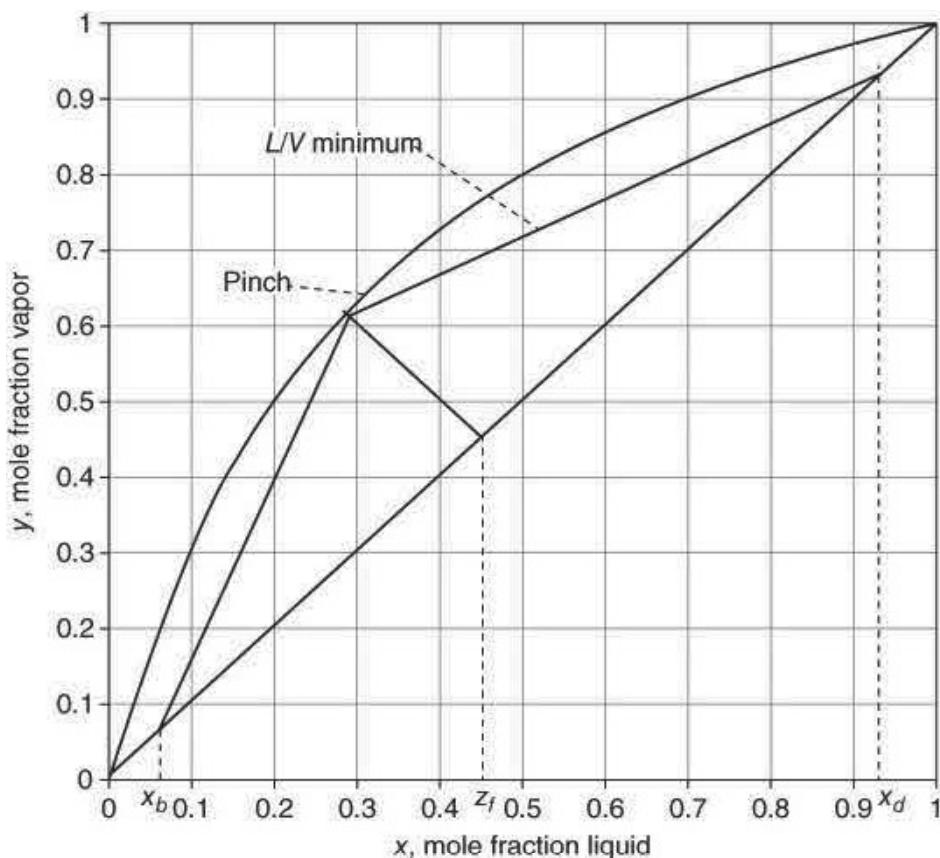
For example, if  $H_F$  is a saturated liquid, the value of  $q$  is zero, which represents a horizontal line on the McCabe–Thiele diagram.



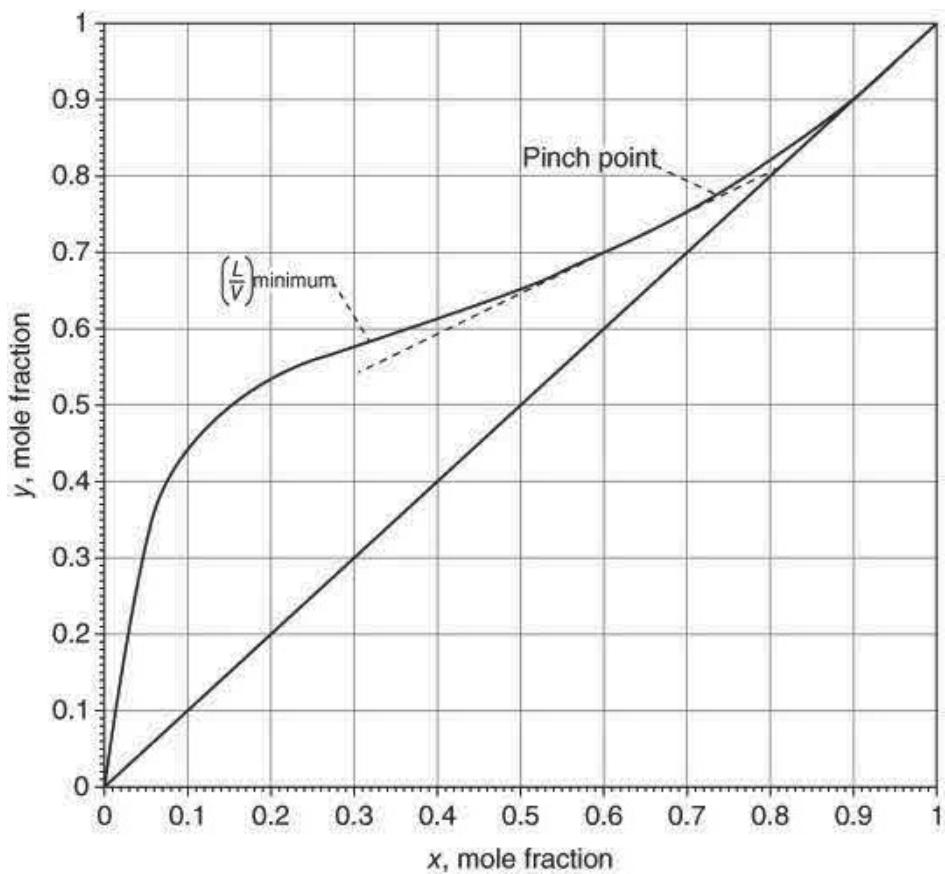
**Figure 11.2** A McCabe–Thiele diagram.

The number of theoretical stages is obtained by drawing a vertical line from an operating line to the equilibrium curve. This is equivalent to finding the equilibrium compositions for a stage. A horizontal line from the equilibrium curve back to the operating line gives the compositions of the passing streams, that is, the material balance. This is repeated from one end of the column to the other.

An important feature is the ability to determine the minimum reflux ratio,  $L/V$ , which occurs at an infinite number of stages. This is known as pinch point and usually occurs when a line drawn from the overhead composition specification is connected to the feed line– $q$ -line intersection as shown in [Figure 11.3](#). If the equilibrium curve has an unusual shape, such as the methanol–water system a different pinch point may be located as shown in [Figure 11.4](#). A rule of thumb commonly used for the design of distillation columns is that the operating reflux should be between 1.25 and 2.0 times the minimum.

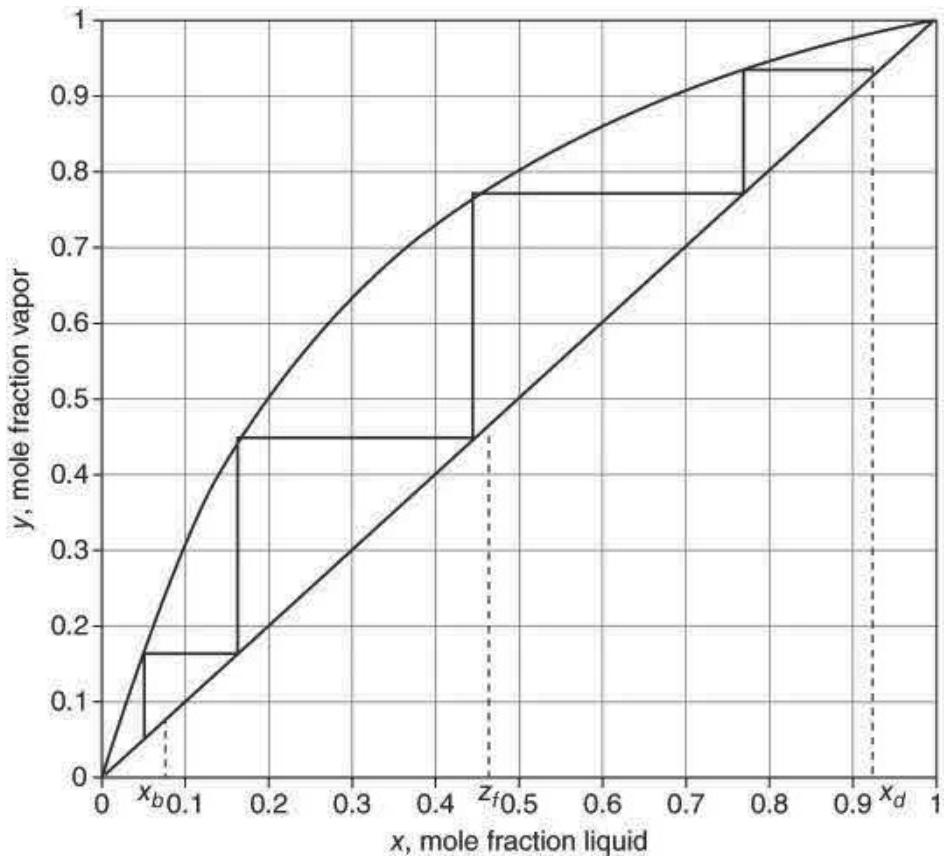


**Figure 11.3** Minimum reflux.



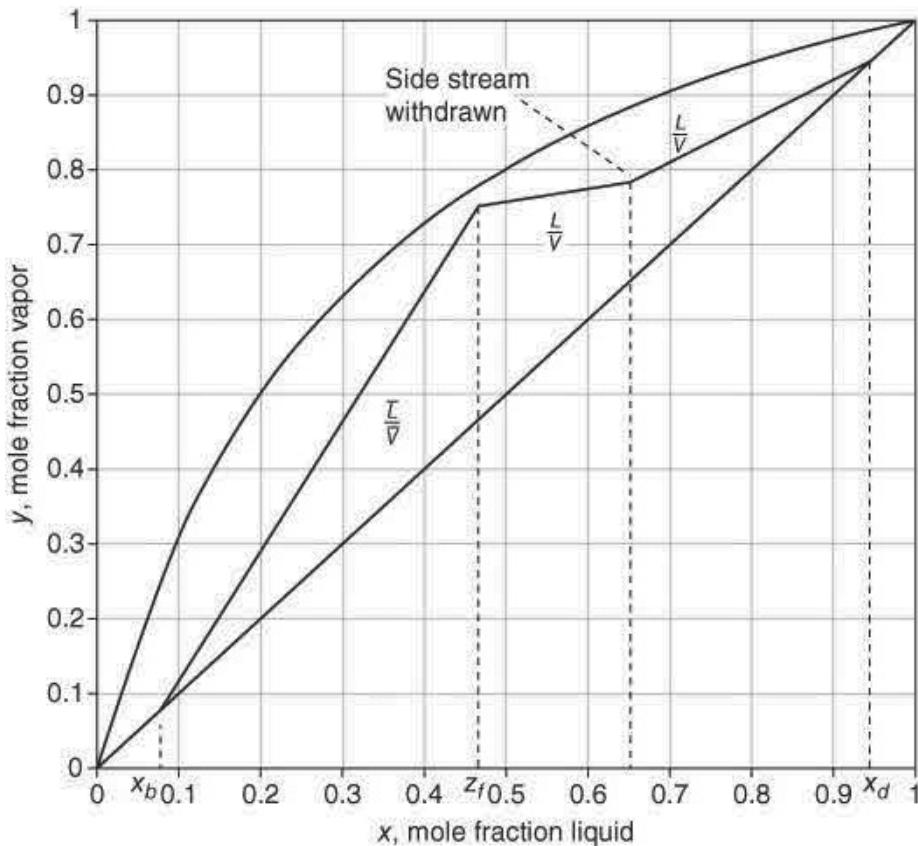
**Figure 11.4** Minimum reflux with nonideal equilibrium.

An estimate of the minimum number of stages required, which occurs at total reflux, is shown in [Figure 11.5](#).



**Figure 11.5** Minimum number of stages.

If a side stream is required, a third operating line is introduced as shown in [Figure 11.6](#). This operating line starts at the feed– $q$ -line intersection, and has a slope  $L'/V'$ . After



**Figure 11.6** Operating lines with a side stream.

determining the operating reflux ratio a McCabe–Thiele diagram can be constructed and theoretical stages stepped off as described above. [Section 11.3](#) gives a detailed McCabe–Thiele example.

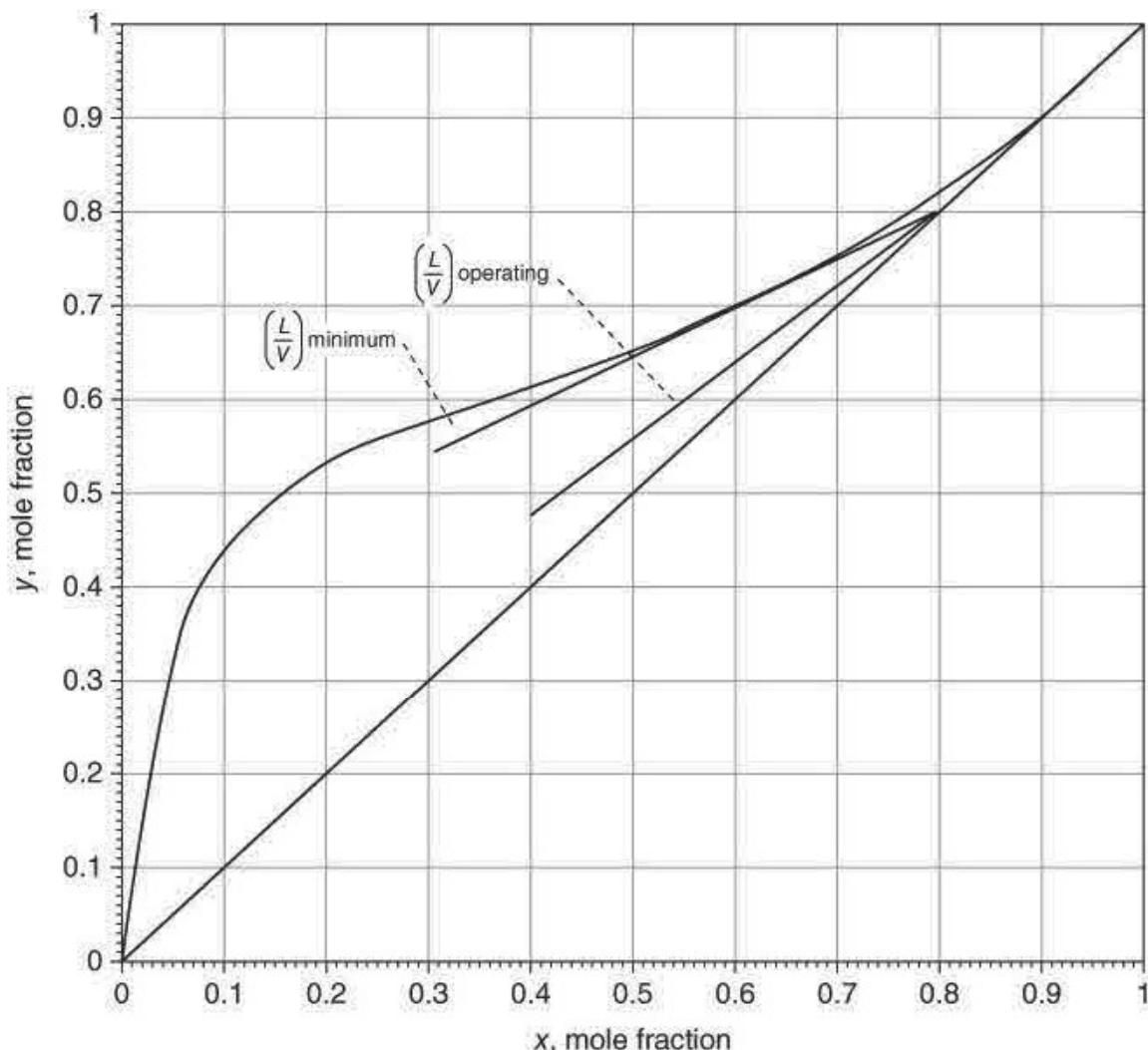
## 11.4 A Three-Product Distillation Example

A 20 mol% ethanol–water solution is to be distilled. Two products 80 mol% ethanol and 40 mol% ethanol are required. The feed is available as a saturated liquid. The composition of the bottom product should not exceed 0.25 mol% ethanol. The external reflux ratio is to be four times the minimum. The shape of the equilibrium curve necessitates a relatively high reflux ratio. The side stream draw-off is 10 lbmol/h. Using a basis of 100 lbmol/h, determine the number of theoretical stages required, the feed location, the side stream location, and the quantities of the products. The experimental data of Kirschbaum and F. Gerstner (1939) is to be used. The data is plotted in [Figure 11.7](#) as is the minimum  $L/V$  of 0.5, which corresponds to a minimum external reflux ratio  $L/D$  of 1.0. The enriching section operating line's  $L/V$  of 0.8 is derived from the external reflux ratio specification, which is also shown and corresponds to an operating external reflux ratio of 4. An overall material balance yields the following results:

$$\text{Distillate } D = 24.8 \text{ lbmol/h}, x_D = 0.8$$

$$\text{Side stream } S = 10 \text{ lbmol/h}, x_S = 0.4$$

Bottoms  $B = 65.2 \text{ lbmol/h}$ ,  $x_B = 0.25$



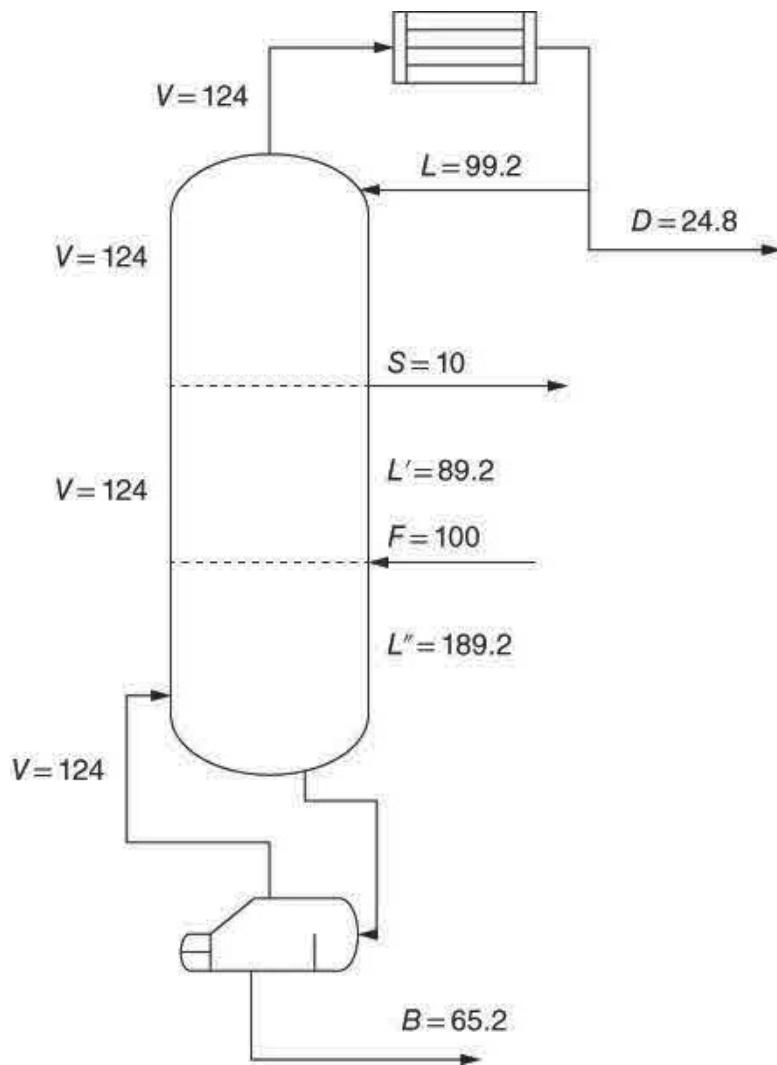
**Figure 11.7**  $L/V$  minimum and operating.

The various liquid and vapor flow rates throughout the column, based on equal molar overflow, are given in [Figure 11.8](#), from which the operating line slopes are derived, which are shown as follows.

Enriching section  $L/V = 99.2/124 = 0.8$

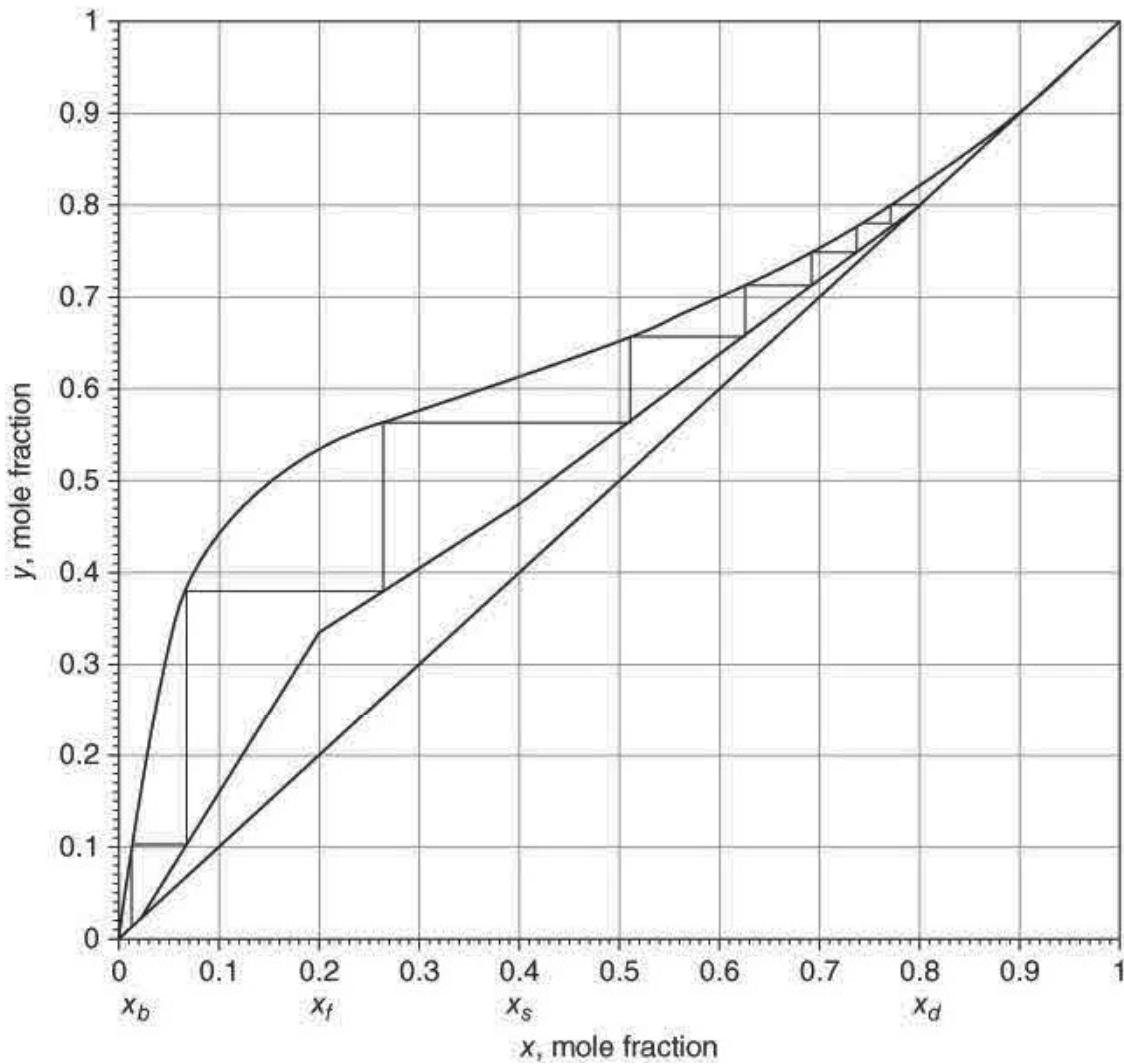
Side stream section  $L'/V' = 89.2/124 = 0.72$

Stripping section  $L''/V'' = 189.2/124 = 1.53$



**Figure 11.8** Three product distillation example with equal molal overflow.

The preliminary design is given in [Figure 11.9](#). There are a total of eight theoretical equilibrium contacts, one of which is the reboiler, as well as a total condenser. The feed is on stage 7 and the side stream is on stage 6, counting from the top down. This is one of several possible solutions, since there is a trade-off between the number of stages, the reflux ratio, equipment cost, and energy considerations.



**Figure 11.9** Preliminary design – three product distillation example.

## 11.5 Preliminary Design and Rating Models

### 11.5.1 DSTWU

The DSTWU model is applicable to both binary and multicomponent systems and was developed for systems that exhibit approximately constant relative volatility. The DSTWU model is based on the Gilliland (1940) correlation shown in [Figure 11.10](#). This correlation was developed from the operating data of many columns and relates the minimum and operating reflux ratios  $R_{min}$  and  $R$  to the minimum and actual number of theoretical stages  $N_{min}$  and  $N$ . Gilliland's data was correlated by Molokanov et al. (1972), which resulted in an analytical version of the Gilliland correlation, [Equation 11.8](#).

$$\frac{N - N_{min}}{N + 1} = 1 - \exp \left[ \left( \frac{1 + 54.4X}{11 + 117.2X} \right) \left( \frac{X - 1}{X^{0.5}} \right) \right] \quad \underline{\text{11.8}}$$

where

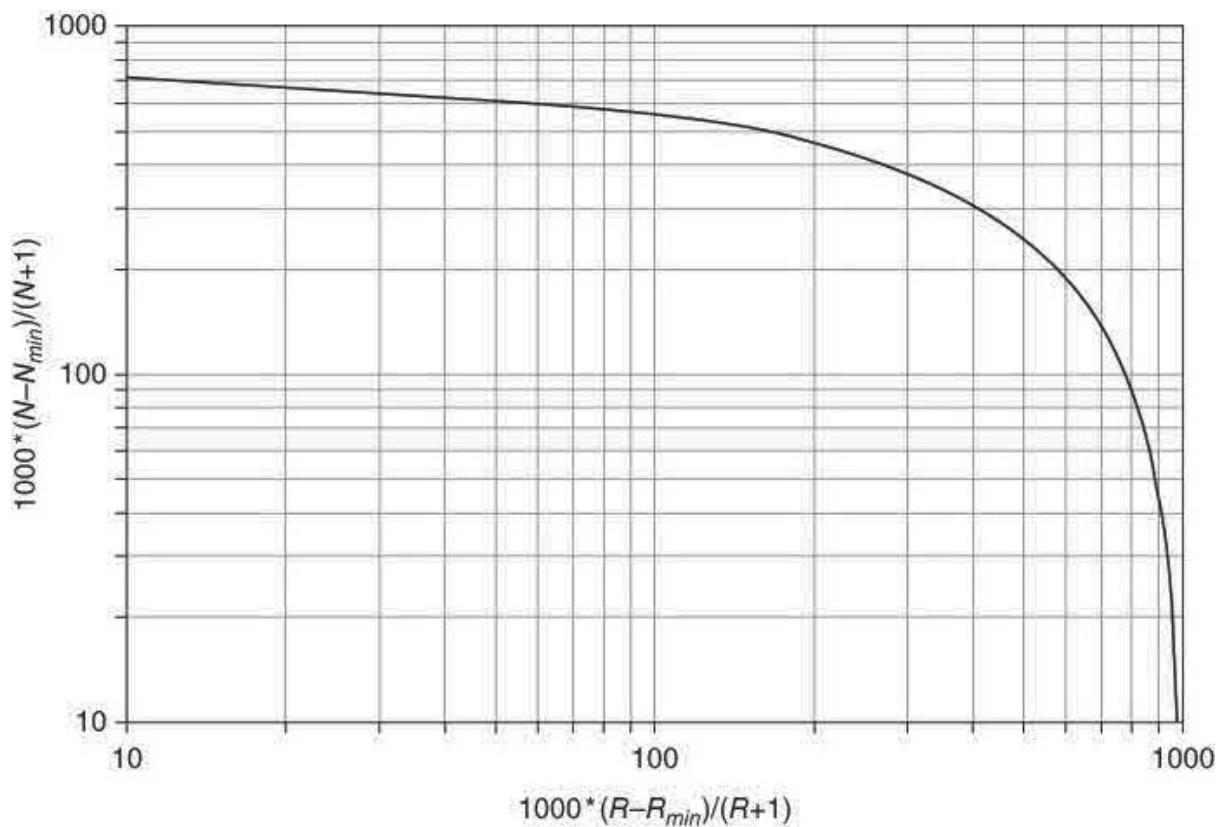
$$X = \frac{R - R_{min}}{R + 1}$$

The minimum reflux ratio is obtained by applying Underwood's (1946) method, which is applicable to binary or multicomponent systems. In the latter case, it is necessary to select light and heavy key components and solve for the roots of Equation 11.9. Here  $\alpha_i$  is the relative volatility of component  $i$ ,  $z_i$  is the mole fraction in the feed of component  $i$ ,  $q$  is the thermal state of the feed, using the McCabe–Thiele definition, and  $\theta$  is a root of the equation. The value of  $\theta$ , which lies between the light and heavy key components, is substituted into Equation 11.10 to calculate the minimum reflux ratio.

$$\sum_i \frac{\alpha_i z_i}{\alpha_i - \theta} = 1 - q \quad \underline{\text{11.9}}$$

$$R_{min} + 1 = \sum_i \frac{\alpha_i x_i^D}{\alpha_i - \theta} \quad \underline{\text{11.10}}$$

where  $x_i^D$  refers to the mole fraction of component  $i$  in the distillate.



**Figure 11.10** The Gilliland correlation.

The minimum number of stages is calculated with Winn (1958) equation, an improvement on Fenske's (1932) equation where the usual definition of relative volatility is replaced by

$$K_i = \beta_i (K_R)^{\theta_i}$$

**11.11**

and the parameters  $\beta_i$  and  $\theta_i$  are determined from the equilibrium data for each component. Winn's equation is

$$N_{min} = \frac{\ln \left[ \left( \frac{x_D}{x_B} \right)_{LK} \left( \frac{x_B}{x_D} \right)_{HK}^{\theta_{LK}} \right]}{\ln \beta_{LK}} \quad \text{11.12}$$

The procedure for estimating the distribution of nonkey components is as follows. Assuming that [Equation 11.13](#) applies where  $d_i$ ,  $b_i$ ,  $d_r$ , and  $b_r$  are the

$$\left( \frac{d_i}{b_i} \right) = \left( \frac{d_r}{b_r} \right) \alpha_{i,r} \quad \text{11.13}$$

distillate and bottoms componential flows of components  $i$  and  $r$ , and  $\alpha_{i,r}$  is their relative volatility, and when [Equation 11.13](#) is combined with the overall componential material balance, [Equation 11.14](#), where  $f_i$  is the feed of component  $i$ , [Equations 11.15a](#) and b are obtained.

$$f_i = d_i + b_i \quad \text{11.14}$$

$$b_i = \frac{f_i}{1 + \left( \frac{d_r}{b_r} \right) \alpha_{i,r}} \quad \text{11.15a}$$

$$d_i = \frac{f_i \left( \frac{d_r}{b_r} \right) \alpha_{i,r}}{1 + \left( \frac{d_r}{b_r} \right) \alpha_{i,r}} \quad \text{11.15b}$$

An example of the use of DSTWU can be found in Chapter Eleven Examples/DSTWU Example. The model's primary input is shown in [Figure 11.11](#). Selection of the Calculation Options tab enables a user to specify a table of reflux versus the number of theoretical stages.

Main Flowsheet **B1 (DSTWU) - Input** +

Specifications     Calculation Options    Convergence    Information

**Column specifications**

Number of stages: **10**

Reflux ratio: **6**

**Pressure**

Condenser: **14.7** psia

Reboiler: **14.7** psia

**Key component recoveries**

Light key:

Comp: **METHANOL**

Recov: **0.95**

Heavy key:

Comp: **ETHANOL**

Recov: **0.05**

**Condenser specifications**

Total condenser

Partial condenser with all vapor distillate

Partial condenser with vapor and liquid distillate

Distillate vapor fraction: **0**

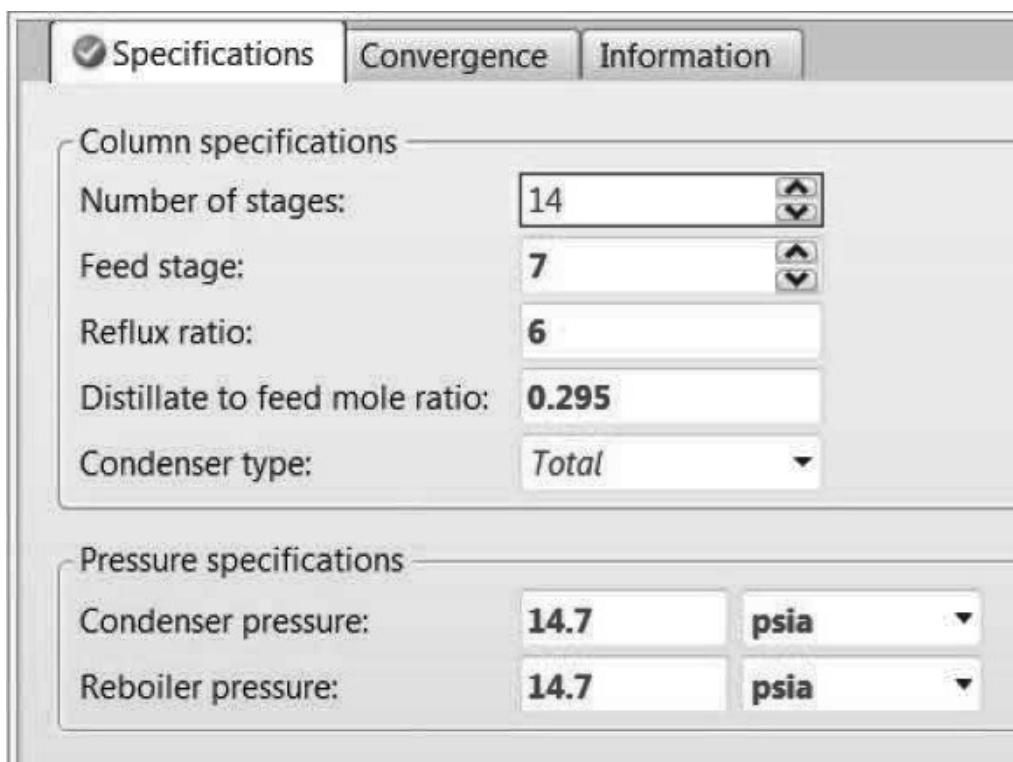
This screenshot shows the 'B1 (DSTWU) - Input' window of a software interface. The window has tabs at the top: 'Specifications' (checked), 'Calculation Options' (checked), 'Convergence', and 'Information'. Under 'Specifications', there are sections for 'Column specifications', 'Pressure', 'Key component recoveries', and 'Condenser specifications'. In 'Column specifications', the 'Number of stages' is set to 10 and 'Reflux ratio' is set to 6. Under 'Pressure', both 'Condenser' and 'Reboiler' pressures are set to 14.7 psia. In 'Key component recoveries', 'METHANOL' is designated as the light key with a recovery of 0.95, and 'ETHANOL' is designated as the heavy key with a recovery of 0.05. Under 'Condenser specifications', the 'Total condenser' option is selected. A 'Distillate vapor fraction' field is also present with a value of 0.

**Figure 11.11** DSTWU primary input.

## 11.5.2 Distl

The Distl model is applicable to both binary and multicomponent systems and makes use of the equal molal overflow and constant relative volatility approximations. The model employs Edmister's method and rates single-feed, two-product columns. Details can be found in Seader and Henley (1998).

The primary input form is shown in [11.12](#). The DSTWU example was used as input to the Distl model. Results are given in Examples/DistlExample.

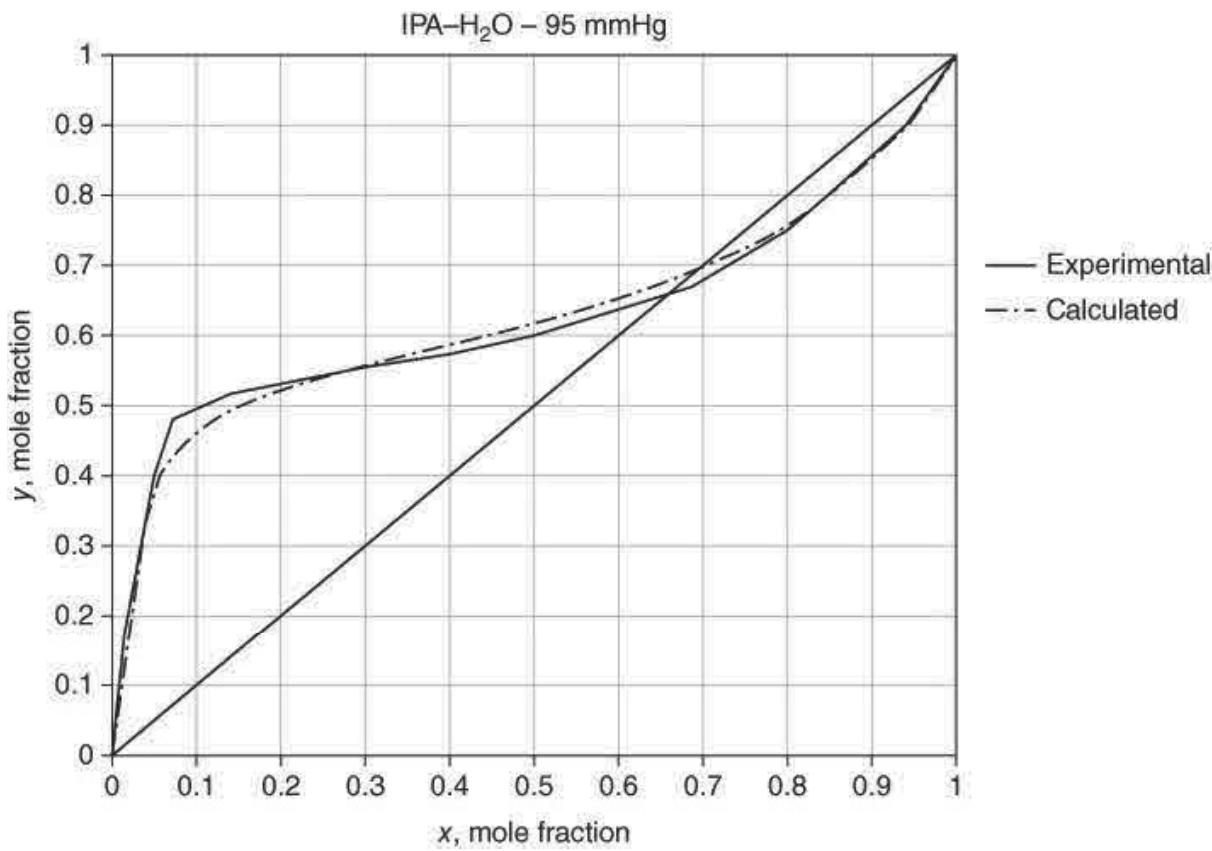


**Figure 11.12** Distl primary input.

## 11.6 Rigorous Models

After developing a preliminary design, it should be possible to develop a rating model of the system of interest. If the system is nearly ideal, or the phases can be represented by an equation of state, the Aspen Plus database is probably suitable. If, on the other hand, the system is sufficiently nonideal that an activity coefficient equation is required, it is advisable to assess the equilibrium data within Aspen Plus by comparing it with experimental data. If the data does not compare favorably, it will be necessary to locate and fit suitable data to an activity coefficient model. If the Aspen Plus database is lacking, one can acquire suitable equilibrium data by either searching the literature, referring to NIST-TDE, developing it experimentally, or estimating it using Unifac. Once suitable data has been found, it should be fit to an activity coefficient model.

As an example, the vapor–liquid equilibrium data for the system isopropanol–water at 95 mmHg of Wilson and Simons (1952) is to be considered for a distillation application. The experimental data was compared against results calculated using a property analysis run “points along a flash curve” using Aspen Plus’s stored data fit with the Wilson equation. [Figure 11.13](#) shows a plot of both data sets.



**Figure 11.13** Experimental versus Wilson equation data.

The usefulness of Aspen Plus's stored data should be assessed by an engineer in light of the precision required for the application. For example, if the application required that the product composition not be in the region approaching the azeotrope, the Aspen Plus data would suffice since the calculated data is hardly different than the experimental data. If, however, the proposed separation required that the product be near the composition of the azeotrope illustrated by Figure 11.13, which shows that the calculated azeotrope composition is about 0.05 mole fraction higher than the experimental value. This would produce serious problems if this column was part of an azeotropic distillation scheme involving two columns operating at different pressures. The quality of the 95 mmHg data calculated from the stored Wilson parameters is suitable since the data that was fit ranges between 77 and 212 °F, as can be seen from Figure 11.14, and the experimental data ranges from a low of about 35 °C (95 °F) to a high of 49 °C (120 °C), but unfortunately does not accurately model the composition of the azeotrope.

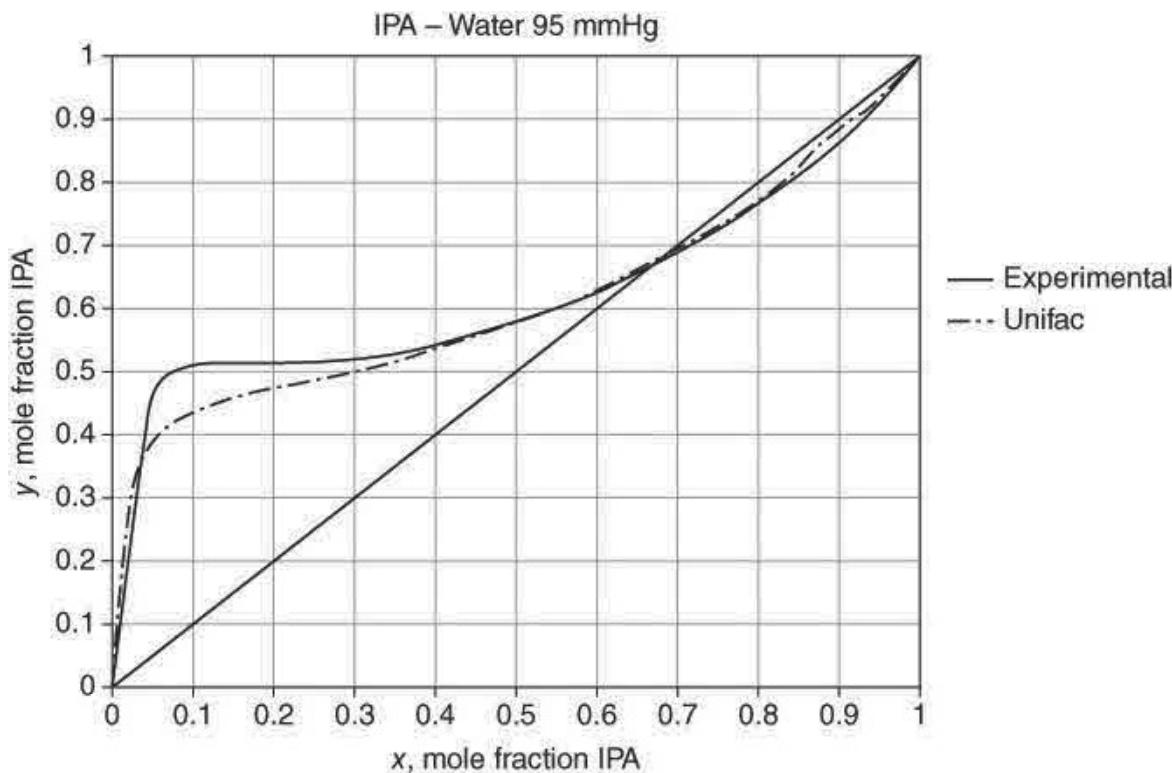
Parameter: WILSON      Data set: 1

Temperature-dependent binary parameters

Component j	WATER
Temperature units	F
Source	APV80 VLE-IG
Property units	
AIJ	-6.2261
AIJ	-0.0133
BIJ	2418.58
BJI	-239.265
CIJ	0
CJI	0
DIJ	0
DJI	0
TLOWER	77
TUPPER	212
EIJ	0
EJI	0

**Figure 11.14** Stored isopropyl alcohol–water Wilson parameters.

If there is no experimental data available for a system, or it cannot be developed in a timely manner, the UNIFAC group contribution may be used to estimate the data. Though this data may be used for preliminary design, it would be wise to set in motion means for experimental verification. For example, [Figure 11.15](#) shows a comparison of experimental and Unifac estimated vapor–liquid equilibrium for the system isopropyl alcohol–water at 95 mmHg. The prediction of the azeotrope composition is remarkable, but the lower composition range is acceptable at best, but again, the use of this data is application driven.



**Figure 11.15** Experimental versus Unifac data.

### 11.6.1 RadFrac

RadFrac is the rigorous distillation modeling workhorse of Aspen Plus. It is based on the inside-out formulation of Boston and Sullivan (1974), which employs a reorganization of the basic equations and the sequence of calculation. A detailed description of the method can be found in Seader and Henley (1998). For problems in which convergence difficulties are experienced, five variations of the basic algorithm are available.

The first example of RadFrac usage here is the same used for DSTWU and Distl. Since RadFrac is a rating or simulation program, the necessary specifications are obtained from the results of the DSTWU example, that is, feed stage location, reflux ratio, and distillate rate. The primary input form for RadFrac is shown in [Figure 11.16](#). Most of the remaining process specifications are straightforward and accessed from the available tabs. The tabs are Streams, in which the feeds and products are specified; Pressure, in which the stage pressures are specified; Condenser, in which the condenser details are specified; and Reboiler, in which the reboiler details are specified. If in [Figure 11.16](#) the reboiler specified is thermosyphon, the Thermosyphon Config and Reboiler tabs become available. When the Thermosyphon Config tab is selected, the thermosyphon reboiler baffle configuration may be selected as shown in [Figure 11.17](#). When the tab Reboiler specifications is chosen, [Figure 11.18](#) appears. This permits various reboiler specifications for either kettle or thermosyphon reboilers to be made. If the Reboiler Wizard button is clicked, the Reboiler Wizard display, on the right side of [Figure](#)

11.18, appears and enables details of an associated heat exchanger to be entered, including a link to the Heatx block. Results for this simulation can be found in Example/RadFracExample1.

The screenshot shows the RadFrac software interface with the following settings:

**Setup options**

- Calculation type: Equilibrium
- Number of stages: 15
- Condenser: Total
- Reboiler: Kettle
- Valid phases: Vapor-Liquid
- Convergence: Standard

**Operating specifications**

- Distillate rate: 162 lbmol/hr (Mole basis)
- Reflux ratio: 6 (Mole basis)
- Free water reflux ratio: 0

**Figure 11.16** Primary RadFrac input.

Configuration    Streams    Pressure    Condenser    Reboiler    3-Phase    Information

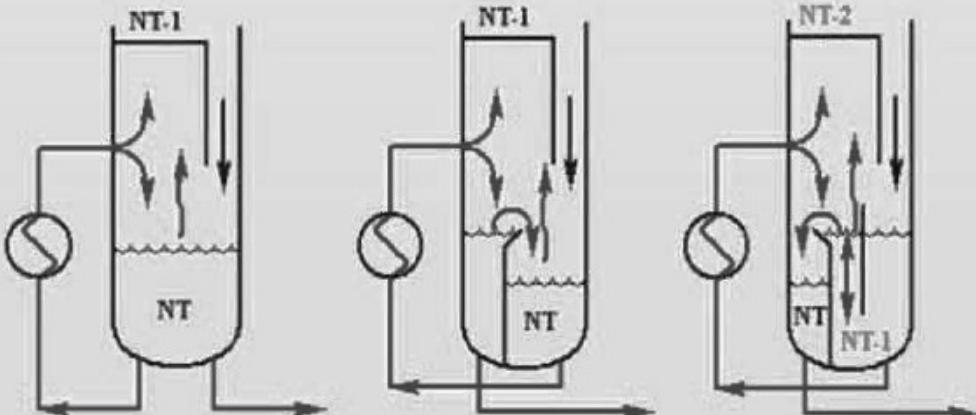
Specify reboiler flow rate  
 Specify reboiler outlet condition  
 Specify both flow and outlet condition

**Flow rate**  
 Mole  

**Outlet condition**  
 Temperature  

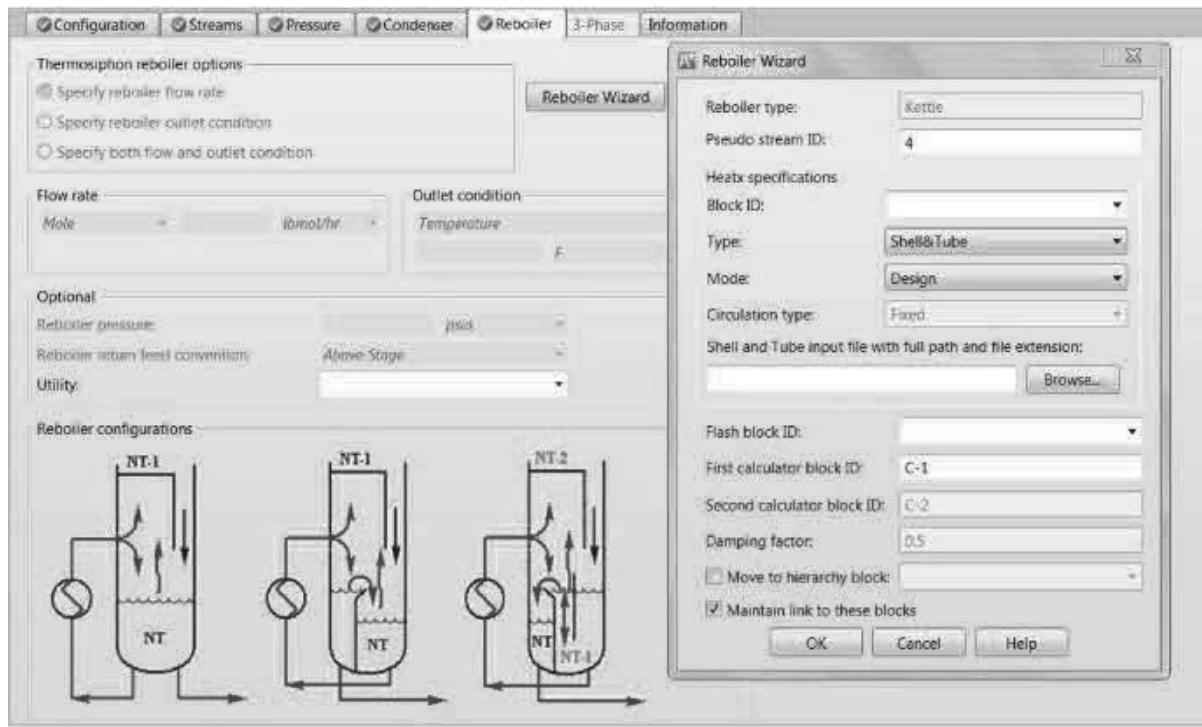
**Optional**  
 Reboiler pressure:     
 Reboiler return feed convention:     
 Utility:  

**Reboiler configurations**



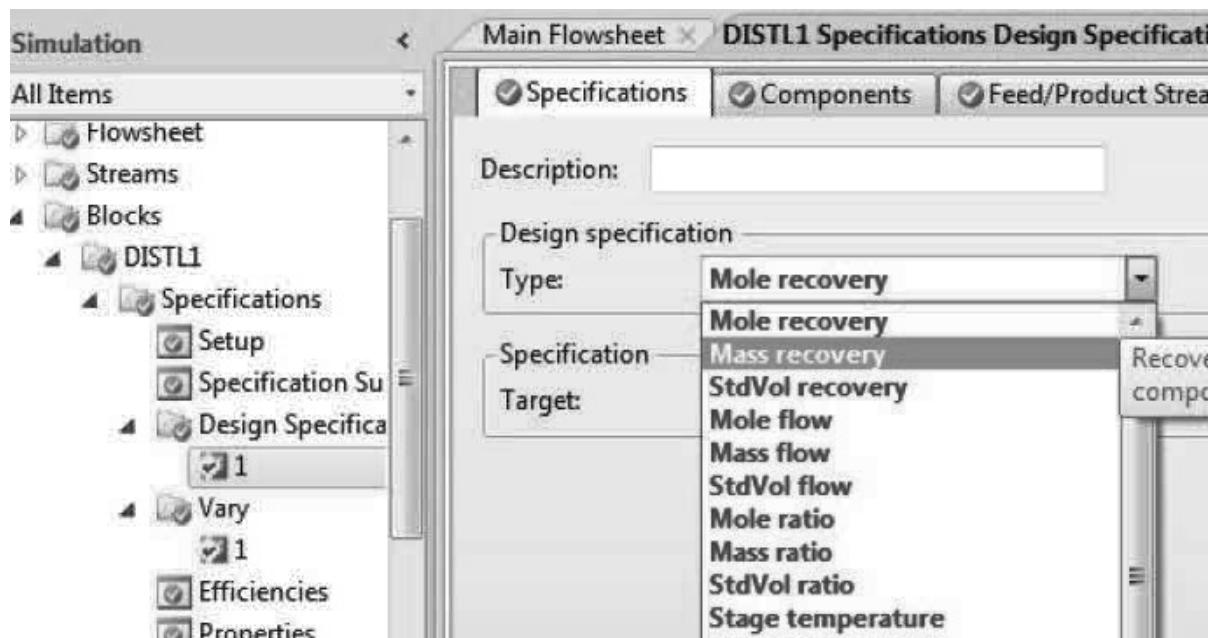
Circulation without baffle    Circulation with baffle    Circulation with auxiliary baffle

**Figure 11.17** Thermosyphon reboiler options.

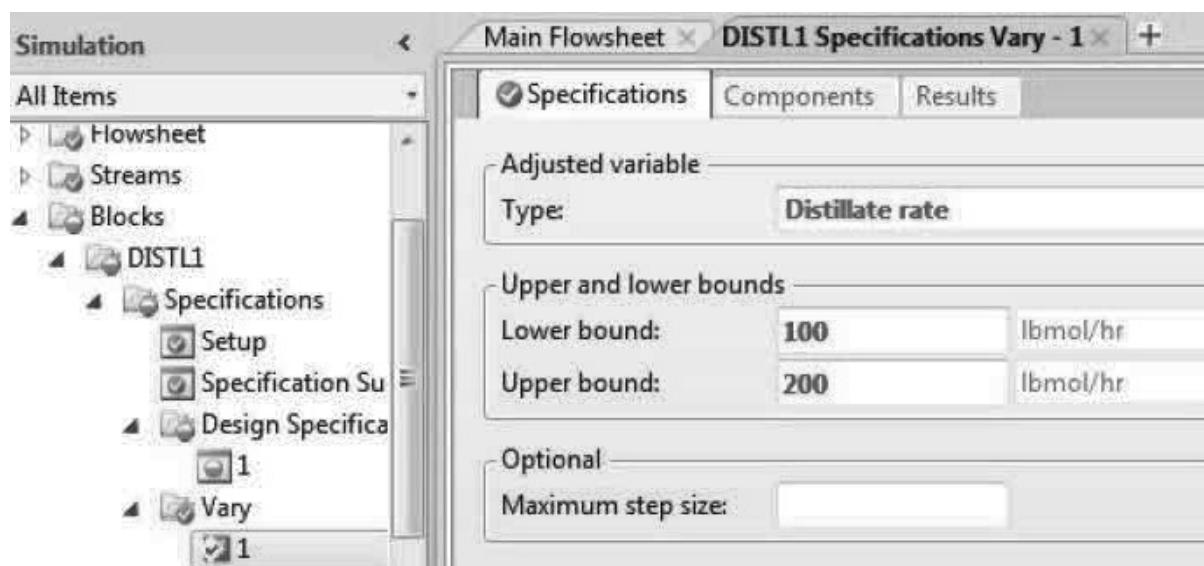


**Figure 11.18** Thermosiphon reboiler specifications and wizard.

The RadFrac block has a design specification function associated with it. In the aforementioned example, the DSTWU solution shows a 0.95 recovery fraction of methanol; however, the rigorous RadFrac solution shows that the recovery is 0.937. One possibility for improving the recovery is to vary a column specification, in this case either the distillate rate or reflux ratio. The use of the Design Specification, varying the distillate rate, is illustrated in RadFracExample2. [Figure 11.16](#) shows a list of options under the configured block named Distl1. The entry Design Specs, followed by Object Manager, New, and Mole recovery target of 0.95 will create the specification, for which the example is shown in [Figure 11.19](#). The selection of Components and Feed/Product Streams allows the completion of the specification. [Figure 11.20](#) created by using the entry Vary shows the bounds within which to search for a result. Results are given in Examples/RadFracExample2.



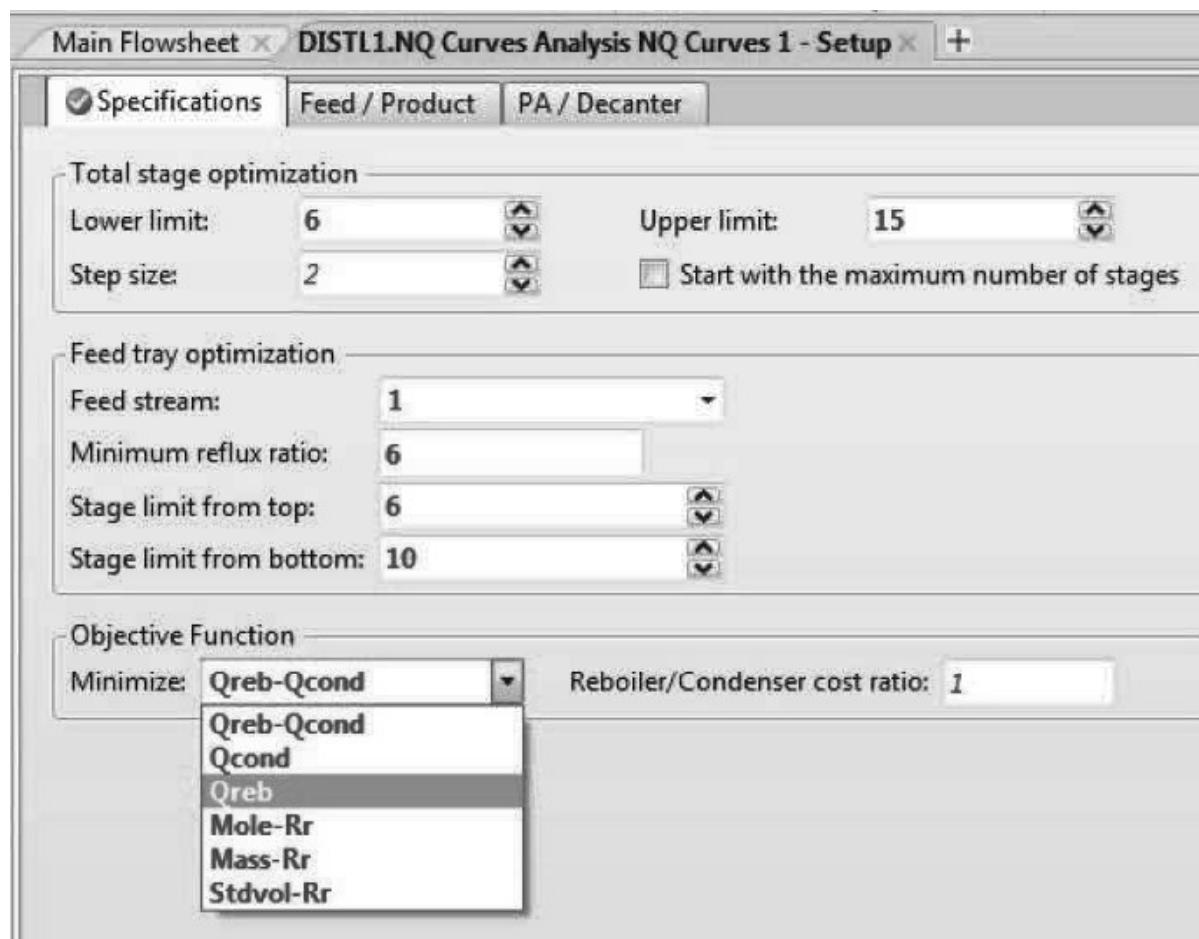
**Figure 11.19** RadFrac design specification.



**Figure 11.20** RadFrac Vary form.

RadFracExample3 illustrates the use of design specifications in conjunction with the normal end specifications of RadFrac. In this case, the distillate rate and reflux ratio are permitted to vary with the aim of achieving composition targets of the distillate and bottoms composition of 0.95 mole fraction methanol and 0.01 mole fraction methanol, respectively. The solution shows that for the limits of the perturbed variables the bottoms composition is obtained and the overhead specification is close and probably acceptable. Note that the Aspen Plus results show many errors but in the practical sense they are meaningless. These results are obtained by using two implementations of the Design Spec and Vary functions. The final values of the reflux ratio and the distillate rate are given in the report.

The remaining design questions are as follows. Are the number of stages near optimal and where is the best feed location? RadFrac addresses these questions by selecting NQ curves under the list of options for the entry Dist1. The setup of this capability is shown in [Figure 11.21](#) and is illustrated in the Examples//Example3a. In this example, many errors, some of which are shown in [Figure 11.22](#), are generated. The various reasons for the errors include limitations at the bounds of perturbed variables. In this case, the optimal feed location and number of stages required were correctly determined from DSTWU and are given in the base case solution. This will usually not occur unless the species involved form relatively ideal mixtures.



**Figure 11.21** NQ curves specifications.

```

Iteration = 2 Feed Stage = 6 Objective Function = 0.37617E+08 J/sec

Convergence iterations:
OL   ML   IL   Err/Tol
1     4    12    73.239
2     2    4     3.9229
3     2    3     0.96662

** ERROR WHILE GENERATING RESULTS FOR UNIT OPERATIONS BLOCK: "DISTL1"
(MODEL: "RADFRAC")
DESIGN SPEC IS NOT SATISFIED BECAUSE ONE OR MORE MANIPULATED
VARIABLE IS AT ITS BOUND.

Iteration = 3 Feed Stage = 4 Objective Function = 0.37618E+08 J/sec

Feed tray optimization converged for NSTAGE = 8
Optimum feed stage = 5 Objective Function = 0.37611E+08 J/sec
* WARNING WHILE GENERATING RESULTS FOR UNIT OPERATIONS BLOCK: "DISTL1"
(MODEL: "RADFRAC")
NQ CURVE "1" STOPPED AT STAGE "8" SINCE THERE IS NO SIGNIFICANT
CHANGE IN OBJECTIVE FUNCTION WHEN INCREASING NUMBER OF STAGES.

** Converging the base case for NSTAGE = 15 **

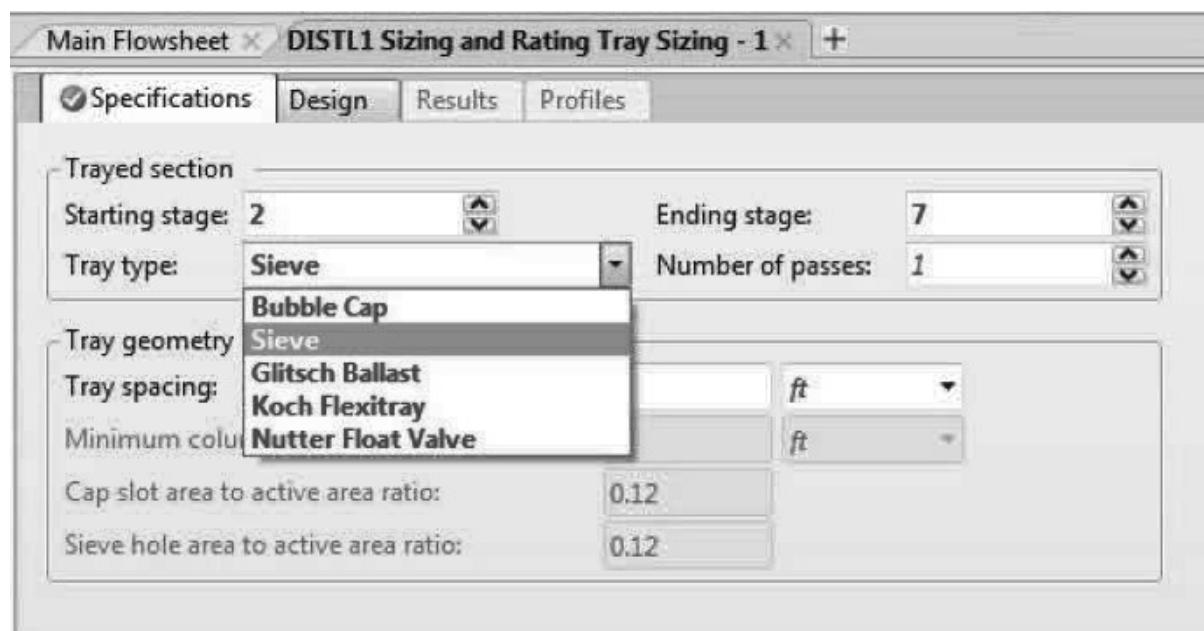
Convergence iterations:
OL   ML   IL   Err/Tol
1     5    14    0.49844E-03

*** END GENERATION OF NQ CURVES ***

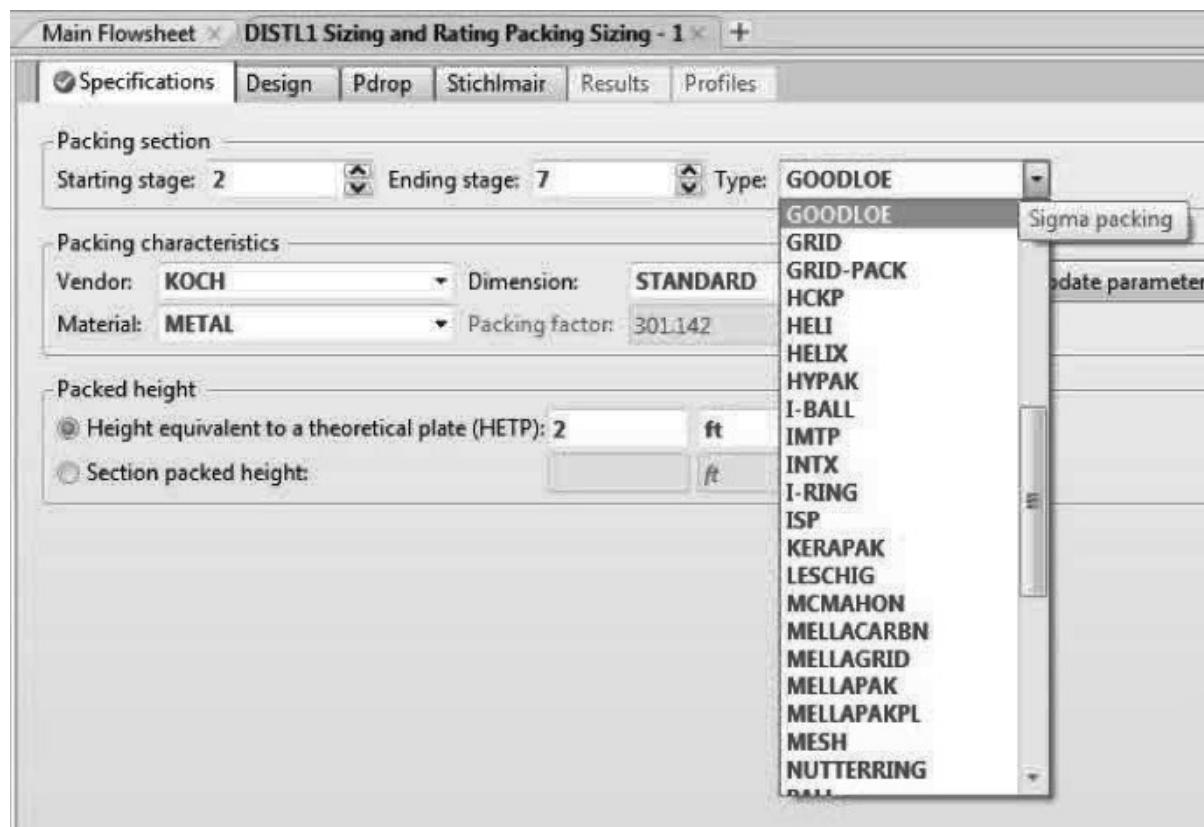
```

**Figure 11.22** Errors generated By NQ run.

Aspen Plus's RadFrac block provides many detailed design functions that are accessible through the Tray and Packing Sizing and Rating folders under the RadFrac block. These functions are based on vendor-provided information or well-known methods described in the literature. Details of the calculations, correlations, and so on can be found in Aspen Plus's Help documentation, for RadFrac, follow links to Appendix A. An example of the tray design input is given in [Figure 11.23](#). Note that five different tray types are available. In this example, the sizing function was used for three sections of the column, that is, enriching, feed, and stripping. Results are shown in Examples/Example3b. When using RadFrac for rating or simulation, the design specifications should be removed and the operating specifications such as reflux ratio and distillate rate should be used. Additionally, the tray or packing design specifications should be removed and replaced with the equivalent tray and packing rating functions. An example of such an implementation is shown in Examples/Example3c. [Figure 11.24](#) shows an example of the packing input.



**Figure 11.23** Tray design input.



**Figure 11.24** Packing input.

## 11.6.2 Extract

Extract is a rigorous extraction column simulation block. The material, energy, and equilibrium equations given in [Equations 11.6](#) apply with the understanding that the V phase nomenclature is fully appropriate for

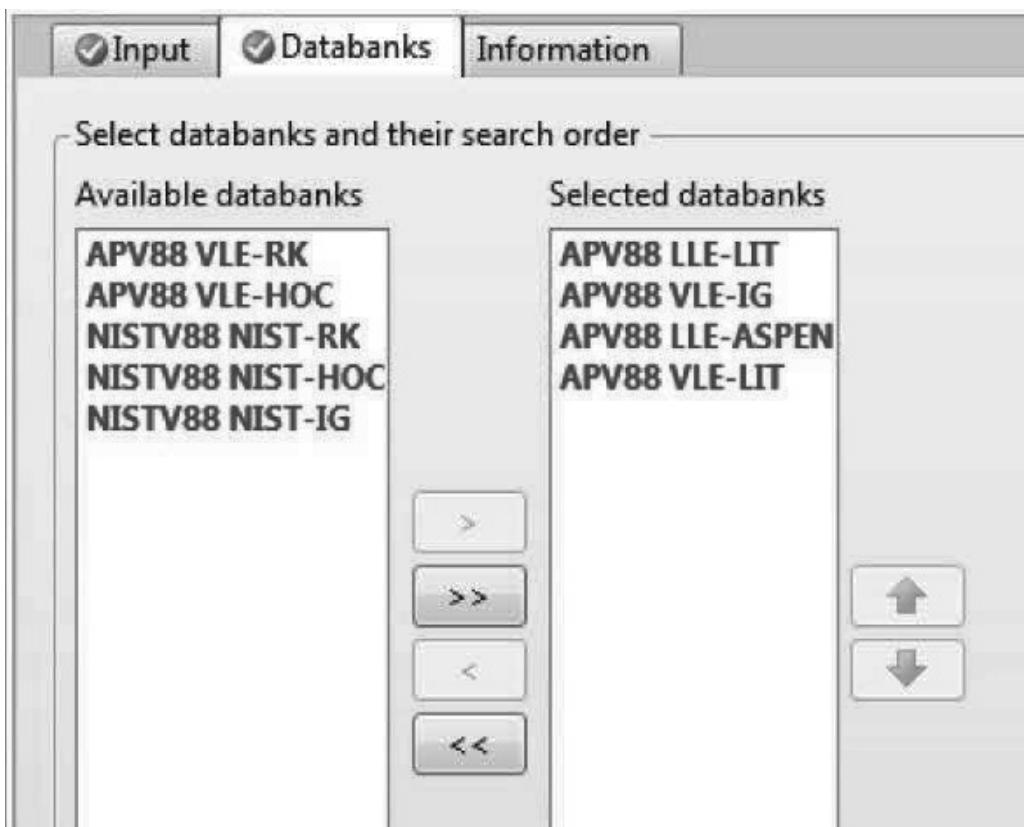
interpretation as the second liquid. Because there are no phase changes, there are rarely large energy swings in extraction columns and thus they operate virtually isothermally. There are no condensers or reboilers, and therefore the number of degrees of freedom is equal to the number of available equations, and no operating specifications are required. The Extract block is a rating or simulation model and its use requires the specification of feed rates, states, and locations as well as the number of equilibrium stages.

When preparing an extraction column simulation, a review of the available data in Aspen Plus's databases is imperative. Figure 11.25 shows the Uniquac parameters for the components of the extraction example given in Examples/ExtractExample. These parameters are located in the subheadings under Parameters/Binary Interaction/UNIQ-1. Figure 11.25 shows that the source of most of these parameters is APV70LLE-LIT (liquid–liquid equilibrium data), but the data for the methanol–water and ethanol–water systems are derived from vapor–liquid equilibrium data. The choice of the source of the data is shown in Figure 11.26. The previous caution about data source applies here. Is quaternary or ternary data available in the literature for this system? Do multicomponent results generated by Aspen Plus's stored data agree with experimental data? Note the temperature limitations on the data in Figure 11.25. Simulation work carried on outside of the applicable temperature ranges of the data has potential for substantial error.

The screenshot shows the Aspen Plus software interface with the 'Input' tab selected. In the center, there is a table titled 'Temperature-dependent binary parameters'. The table has a header row with columns for Component i and Component j, followed by rows for Temperature units, Source, and Property units. Below these are rows for various binary interaction parameters (AII, BII, CII, DII, DJI) and two rows for temperature limits (TLOWER and TUPPER). The data is as follows:

	METHANOL	METHANOL	METHANOL	ETHANOL	ETHANOL	WATER
Component i	METHANOL	METHANOL	METHANOL	ETHANOL	ETHANOL	WATER
Component j	ETHANOL	WATER	TOLUENE	WATER	TOLUENE	TOLUENE
Temperature units	C	C	C	C	C	C
Source	VLE-IG	VLE-IG	LLE-LIT	LLE-LIT	LLE-LIT	LLE-LIT
Property units						
AII	-2.6509	-1.0662	0	0	0	0
AII	1.2891	0.6437	0	0	0	0
BII	651.488	432.879	-178.59	185.3	189.19	-350.21
BII	-273.692	-322.131	-397.58	167.38	-357.03	-950.6
CII	0	0	0	0	0	0
CII	0	0	0	0	0	0
DII	0	0	0	0	0	0
DJI	0	0	0	0	0	0
TLOWER	20	24.99	20	20	20	20
TUPPER	78.4	100	40	40	40	40

**Figure 11.25** Uniquac parameters and data sources.



**Figure 11.26** Data source choices.

## 11.7 BatchSep

Batch distillation is commonly used in processing specialty chemicals and pharmaceuticals and has a substantial role in the chemical industries. Aspen Plus's batch distillation block is BatchSep. The equations describing batch distillation are very similar to those describing steady state distillation; however, the material and energy balances are time varying, and, therefore, differential equations are required. An example of a differential material balance analogous to [Equation 11.3](#) is given in [Equation 11.16](#). Here, the alternative form of the material balance is used with the independent variables being mole fractions and total flows. If  $M^j$  is the volumetric stage holdup, and  $\rho$  is the molar density, [Equation 11.16](#) results:

$$\frac{d(x_i^j \rho M^j)}{dt} = z_i^j F^j + y_i^{j+1} V^{j+1} + x_i^{j-1} L^{j-1} - y_i^j V^j - x_i^j L^j - x_i^j S^j \quad \text{11.16}$$

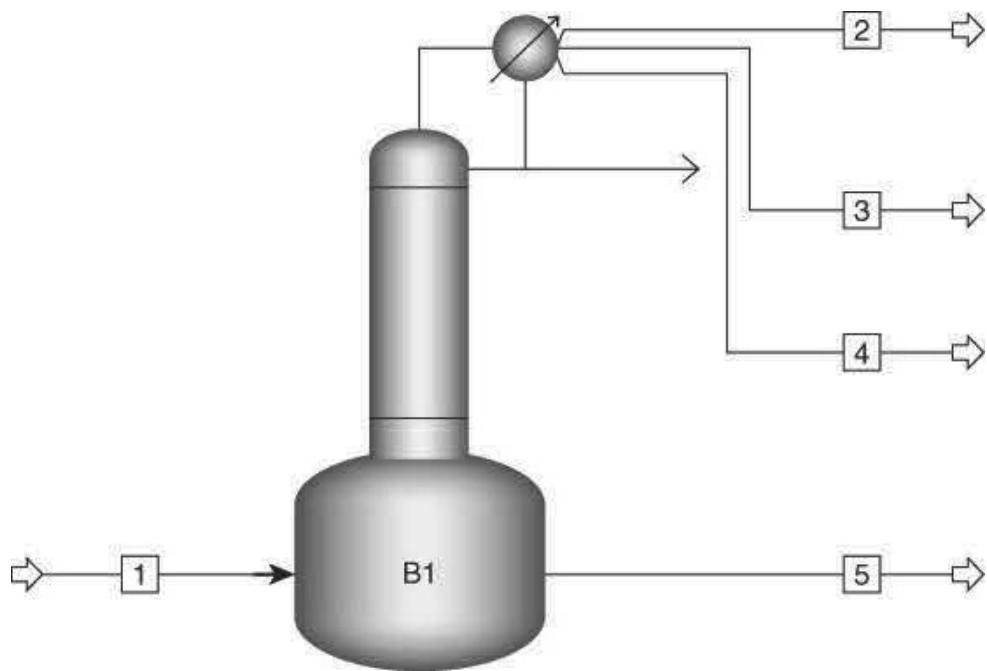
Similarly, a differential energy balance analogous to [Equation 11.6](#) is [Equation 11.17](#).

$$\frac{d(\rho M^j c_p T)}{dt} = H_F^j F^j + H V^{j+1} + h L^{j-1} - H V^j - h L^j - h S^j + Q \quad \text{11.17}$$

For an  $n$  component column, there are  $n$  differential material balances and one differential energy balance per stage. Additionally,  $n$  nonlinear algebraic

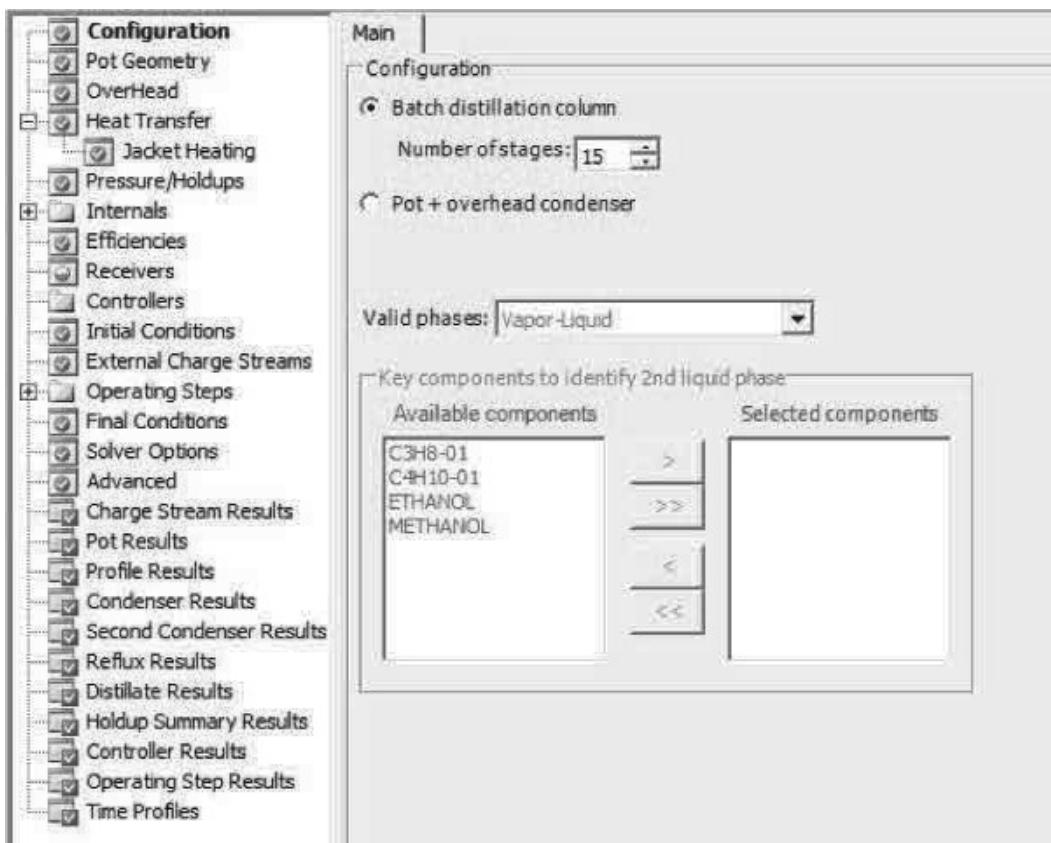
equations describing phase equilibrium and the auxiliary summation of vapor and liquid mole fractions equations are also required per stage. This large set of combined differential and nonlinear equations can be solved numerically. Initial conditions are required for all the variables described in the differential equation terms. Targets during the distillation, such as overhead temperature, are used to identify the beginning and end of a cut, and receivers must be defined for the collection of each of the cuts produced.

An example of a batch distillation application is given in Examples/BatchSepExample. Figure 11.27 shows a schematic of a batch distillation column. Stream 1 is the batch charge, streams 2–4 are the products (cuts), and stream 5 is the pot contents at the end of the distillation. Each of the product streams has a receiver associated with it. BatchSep applications require Aspen Plus release 7.1 or higher.



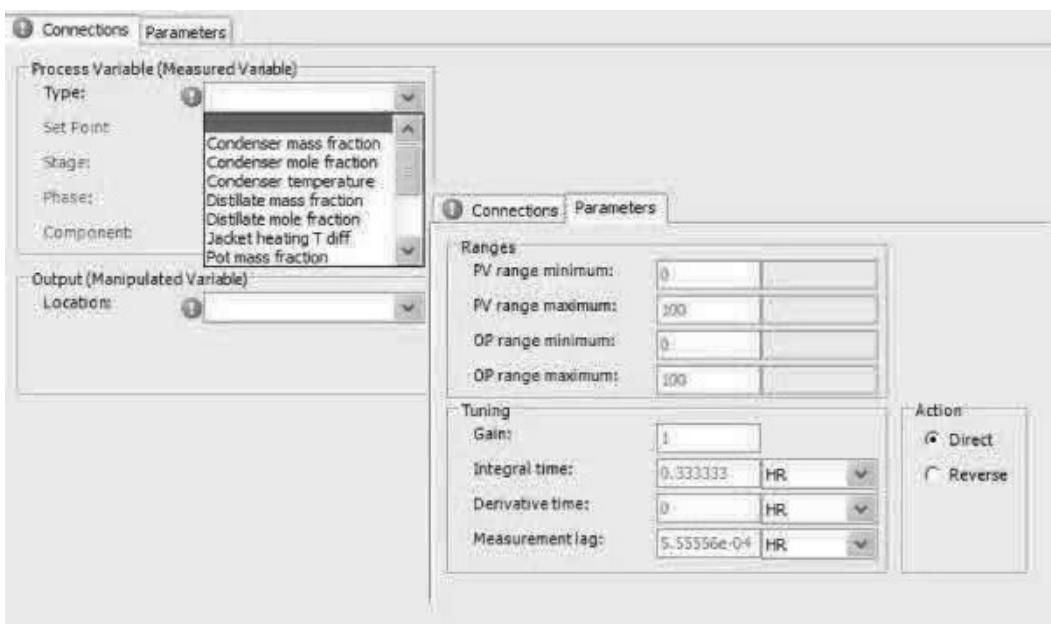
**Figure 11.27** Batch distillation column.

The Setup form for the BatchSep block is shown in Figure 11.28. Note the possibility of two liquid phases in equilibrium with a vapor phase. Each of the available tabs enables the definition of the physical dimensions of the still as well as the column's overhead specification such as reflux ratio. The Jacket Heating tab enables the choice and value of the bottoms specification. The tab Receivers permits the definition of the number of receivers and the association of each product stream with a particular receiver. The Side Draws tab permits the entry of all necessary side draw information.



**Figure 11.28** BatchSep basic setup.

The Controllers folder facilitates the use of a simulated PID type controller during the batch distillation. Figure 11.29 shows the two tabs that permit a user to choose a variable to be controlled and the controller settings that operate the controller. This capability enables one to simulate the real-time behavior of the batch still.



**Figure 11.29** Batch distillation column controller setup.

The input forms for Initial conditions tab are illustrated in [Figure 11.30](#). Both the Main and the Initial Charge tabs are shown.

The screenshot shows two tabs: 'Main' and 'Initial Charge'. The 'Main' tab is selected, displaying the following parameters:

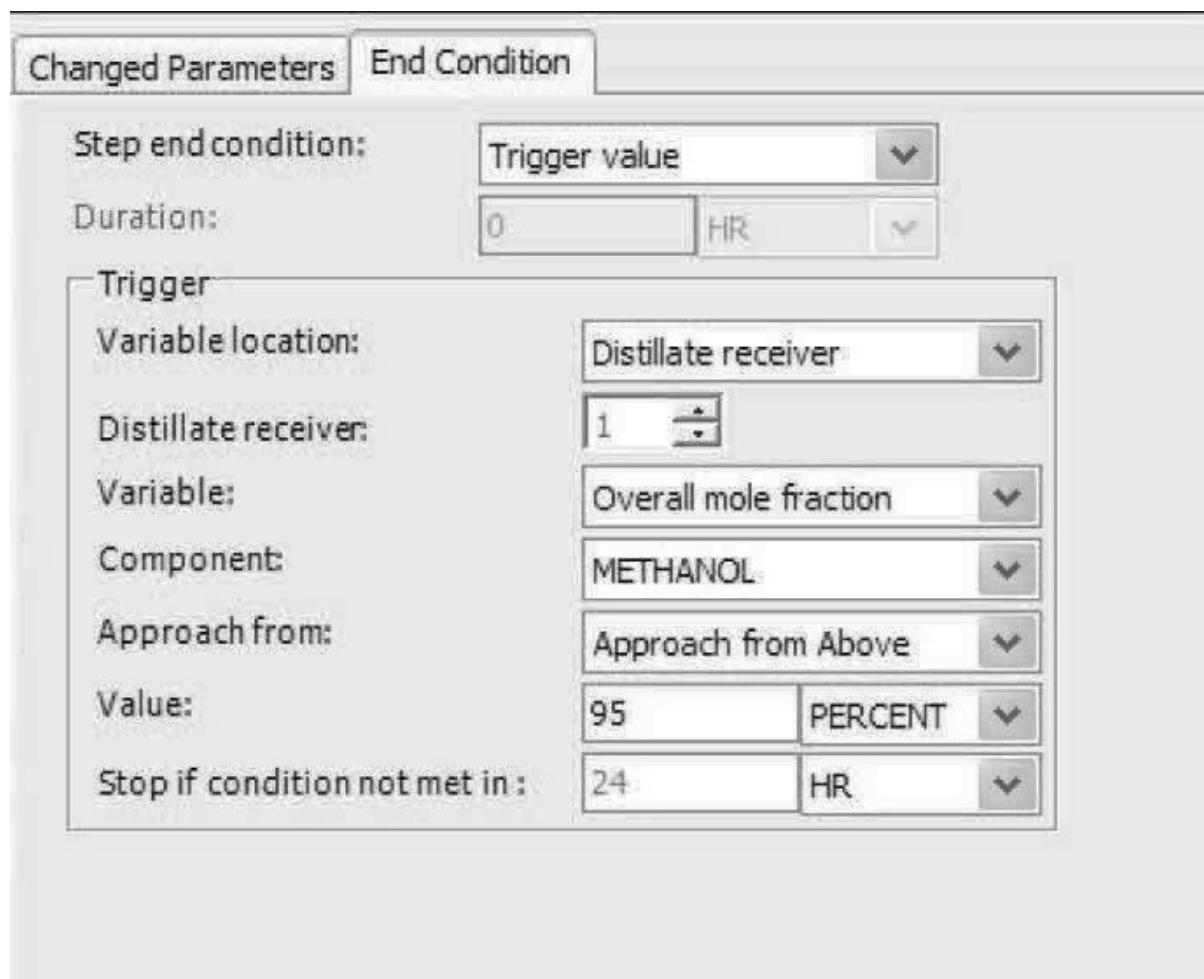
- Initial condition: Total reflux
- Initial drum liquid volumefraction: 0.5
- Initial temperature: 20 °C
- Initial pressure: 760 MMHG

The 'Initial Charge' tab is also visible. It includes the following sections:

- Specify charge time: 1 HR (radio button)
- Specify total charge (radio button, selected)
- Charge basis: Mole (dropdown menu)
- Total initial charge: 550 LBMOL (input field)
- Total fresh charge: 550 LBMOL (input field)

**Figure 11.30** Initial conditions for BatchSep.

Control of the products of the distillation is done with the Operating Steps entry. In this example, there are two operating steps to the process. [Figure 11.31](#) shows the setup of Step One. When all the steps have been executed, the Final Conditions entry permits the choice of disposition of the contents of the reflux drum, the packing, and the pot.



**Figure 11.31** Operating step input – Step one.

## 11.8 Workshops

### Workshop

**11.1a** A mixture of 50 lbmol/h of benzene, 50 lbmol/h of toluene, and 50 lbmol/h of paraxylene is to be distilled at 1 atm. The feed is a saturated liquid at 1 atm. The column is to operate at a reflux ratio of 2.8 and a 1 psi pressure drop across the column is anticipated. 95% of the benzene fed and no more than 5% of the toluene fed is to be taken overhead. Use the shortcut distillation block **DISTWU** to estimate the column performance. Generate a table of reflux ratio versus theoretical stages required for this separation. Create a mixed unit set derived from ENG replacing °F with °C and psi with mmHg for all Workshop 11.1 variations.

### Workshop

**11.1b** Repeat Workshop 11.1a using the physical properties option NRTL. Compare to Workshop 11.1a.

### Workshop

**11.1c** Solve Workshop 11.1a rigorously using RadFrac. Employ 12

theoretical stages with feed on stage 6. Specify that 50 lbmol/h of distillate is required. Solve using the ideal property option. Compare to the results of Workshop 11.1a.

### Workshop

**11.1d** Repeat Workshop 11.1c but use the property option NRTL. Compare to the Workshop 11.1c results.

### Workshop

**11.2a** A mixture of 50 lbmol/h of methanol, 50 lbmol/h of ethanol, and 50 lbmol/h of water is to be distilled at 1 atm. The feed is a saturated liquid at 1 atm. The column is to operate at a reflux ratio of 1.2, and a 1 psi pressure drop across the column is anticipated. 95% of the ethanol fed, and no more than 5% of the water fed, is to be taken overhead. Use the short cut distillation block DISTWU with the physical properties option WILSON to estimate the column performance. Generate a table of reflux ratio versus theoretical stages required for this separation. Create a mixed unit set derived from ENG replacing °F with °C and psi with mmHg for all Workshop 11.2 workshop variations.

### Workshop

**11.2b** Solve Workshop 11.2a rigorously using the block RadFrac. Specify that 50 lbmol/h of bottoms is required. Use 15 theoretical stages with feed on stage 10. Plot the liquid compositions for the column. What do you observe? Compare to the results of Workshop 11.2a.

### Workshop

**11.2c** Repeat Workshop 11.2b rigorously. Specify that 100 lbmol/h of distillate is required. Use 11 theoretical stages with feed on stage 7. Compare to the results of Workshop 11.2b.

### Workshop

**11.3a** The data for this problem (Udoenko and Mazanko, 1964) can be found in the file DataEleven-3bkp in the Workshops folder. Create a set of units, NEWSET, which employs pressure in mmHg and delta  $P$  in atm, copied from the data set MET. Fit the data to the NRTL equation, assigning 0.0 to the  $A_{ij}$  parameters and assigning 0.2 to each  $C$  parameter. Produce a triangular plot of the data.

A mixture of 500 lb/h of benzene and 500 lb/h of 2-propanol is to be extracted with 1000 lb/h of water in a continuous countercurrent extraction column operated at 1 atm and 30 °C. The feeds are liquids at 16 psi and 30 °C. A 1 psi pressure drop across the column is anticipated. The column has four theoretical stages. Use the Extraction block to calculate the column's performance. Create a sensitivity study varying the quantity of water used for extraction from 700 to 1800 lb/h in increments of 100 lb/h. Create a set of units copied from ENG using °C.

## **Workshop**

**11.3b** Use a design specification to determine the quantity of water needed to recover 96% of the alcohol.

## **Workshop**

**11.3c** Use the Decanter block to develop a four-stage batch extraction solution to problem Workshop 11.3b. Divide the amount of water calculated in Workshop 11.3b into four equal quantities so that each can be added to the batch four times. Compare the quantity of alcohol extracted to the solution for the continuous case. What do you conclude?

# **11.9 Workshop Notes**

## **Workshop**

### **11.1a–d**

All results are similar because this system is nearly ideal. Workshop 11.1c has an error message relating to the pressure of the feed stream. Note that the feed pressure, 14.696, is lower than the feed stage pressure. Workshop 11.d's feed is set at 16 psi to fix the problem.

## **Workshop**

### **11.2b**

Results at reflux ratio of 1.2 with a bottoms takeoff of 50 lbmol/h are similar to Workshop 11.2a. [Figure 11.32](#) shows a tabulation of liquid composition as a function of the stage numbers. One may observe that the methanol composition barely changes between stages 6 and 11, which suggests that there are too many stages in the current design.

Stage	MEOH	ETOH	WATER
1	0.415831	0.537867	0.0463019
2	0.306554	0.62654	0.066906
3	0.259327	0.658717	0.0819561
4	0.240209	0.665476	0.0943153
5	0.233316	0.661104	0.10558
6	0.23161	0.651748	0.116642
7	0.232028	0.639896	0.128076
8	0.233254	0.6264	0.140345
9	0.234727	0.611343	0.153929
10	0.23617	0.594407	0.169423
11	0.237405	0.574957	0.187638
12	0.191441	0.606945	0.201615
13	0.151717	0.615114	0.233169
14	0.115867	0.557079	0.327054
15	0.0658543	0.289215	0.64493

**Figure 11.32** Stage compositions.

## Workshop

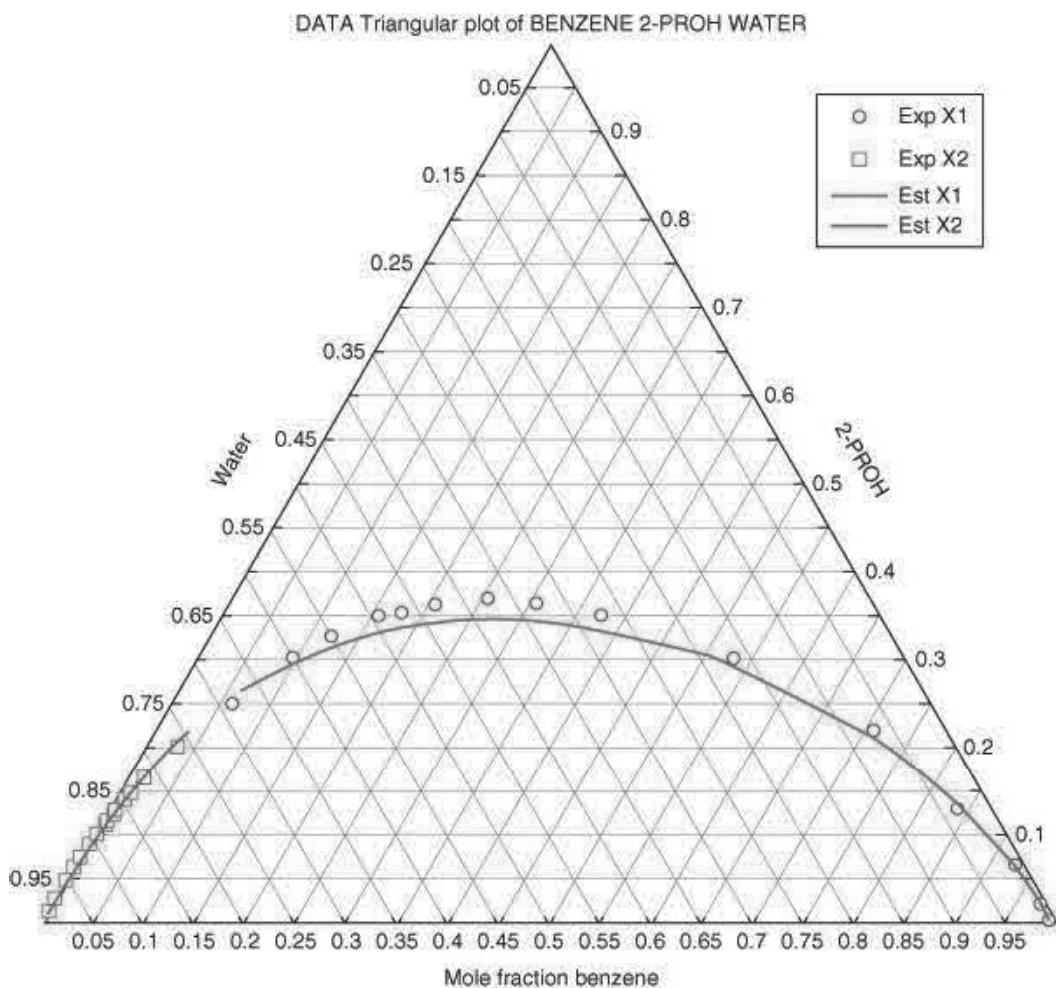
### 11.2c

The column is reconfigured to 11 stages with the feed on stage 7. The results are very close to those of Workshop 11.2c. The column design as proposed by DSTWU has too many stages.

## Workshop

### 11.3a

The triangular plot is shown in [Figure 11.33](#).



**Figure 11.33** Triangular plot.

### Workshop

#### 11.3c

To obtain the total quantity of 2-propanol extracted, it is convenient to setup a Mix block and feed it the aqueous phases from each decanter. When this is first run, one observes that with one quarter of the required water added to the organic feed, no phase splitting occurs. It is necessary to feed both products of the first decanter to the second decanter and then everything proceeds as expected. If an error message referring to insufficient iterations arises, double the default value of 30.

When comparing the results to Workshop 11.3b, one observes that less than half of the alcohol is extracted by batch extraction.

### References

Aspen Plus's Help documentation, Appendix A.

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