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<p>KLM Technology Group P. O. Box 281 Bandar Johor Bahru, 80000 Johor Bahru, Johor, West Malaysia</p>	<p>Kolmetz Handbook of Process Equipment Design</p> <p>Distillation Column Selection, Sizing and Troubleshooting</p> <p>(ENGINEERING DESIGN GUIDELINES)</p>	<p>Co Author:</p> <p>Rev 1 Chew Yin Hoon Rev 2 Ai Li Ling Rev 3 Aprilia Jaya Rev 4 Mochamad Adha Firdaus Rev 5 Reni Mutiara Sari</p> <p>Author / Editor</p> <p>Karl Kolmetz</p>

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INTRODUCTION

Scope

This design guideline covers the basic elements in designing a typical distillation column system, which includes column internals selection and sizing.

In designing a distillation column, the thermodynamics of the vapor and liquid phases must be understood. The vapor-liquid equilibrium (VLE) determines the minimum number of stages required to achieve the degree of separation needed. The minimum reflux ratio also depends on the VLE data of the mixture.

A few equations that are commonly used in the industry are illustrated in this guideline to estimate the minimum number of stages and the minimum reflux ratio of a column based on the VLE data, such as the Fenske-Underwood equation. Some design heuristics are also highlighted. These rules are based on design experiences and take into account both the safety and economical factors.

The selection of column internals is very critical in distillation column design. There is a wide variety of trays and packings in the market. Each design has its strengths and weaknesses. However, the quotations from vendors are sometimes contradictory and confusing. This could lead to a wrong choice of column internals. Therefore, some general considerations are depicted to aid engineers in making the right choice of column internals. In general select trays for high pressure and packings for low pressure.

A distillation column is sized by determining the diameter of the tower. An initial estimation of the tower diameter can be done based on the vapor and liquid loadings in the column.

Today, many technologies present in improving a distillation to obtain less energy and capital saving. Dividing Wall Columns (DWCs) are a promising technology for creating sustainable, more energy and economically efficient processes. A DWC is in essence a fully thermally coupled distillation sequence with only one condenser and one reboiler regardless of the number of products.

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This design guideline also covers what is needed is a method based on sound, credible principles. The theory for the distillation column selection and sizing, dividing wall column and extractive distillation selections. Briefly application, design procedure and technology that typically used in dividing wall column and extractive distillation are also summarized in this guideline.

Included in this guideline is an example of the data sheet used in the industry and a calculation spreadsheet for the engineering design.

Distillation

Distillation is by far the most important separation process in the petroleum and chemical industries. It is the separation of key components in a mixture by the difference in their relative volatility, or boiling points. It is also known as fractional distillation or fractionation.

In most cases, distillation is the most economical separating method for liquid mixtures. However, it can be energy intensive. Distillation can consume more than 50% of a plant's operating energy cost. There are alternatives to distillation process such as solvent extraction, membrane separation or adsorption process. On the other hand, these processes often have higher investment costs. Therefore, distillation remains the main choice in the industry, especially in large-scale applications.

Distillation History

The history of distillation dated back to centuries ago. Forbes has chronicled the full history of distillation in 1948^[1]. Reputedly, it was the Chinese who discovered it during the middle of the Chou dynasty. It was later introduced to India, Arabia, Britain and the rest of the world.

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Early distillation consisted of simple batch stills to produce ethanol. Crude ethanol was placed in a still and heated, and the vapor drawn from the still was condensed for consumption. Lamp oil was later produced using the same method, with crude oil heated in batch stills.

The next progression in the history of distillation was to continually feed the still and recover the light product. Further advancements include placing the stills in series and interchanging the vapor and liquid from each still to improve recovery. This was the first type of counter-current distillation column that we have today.

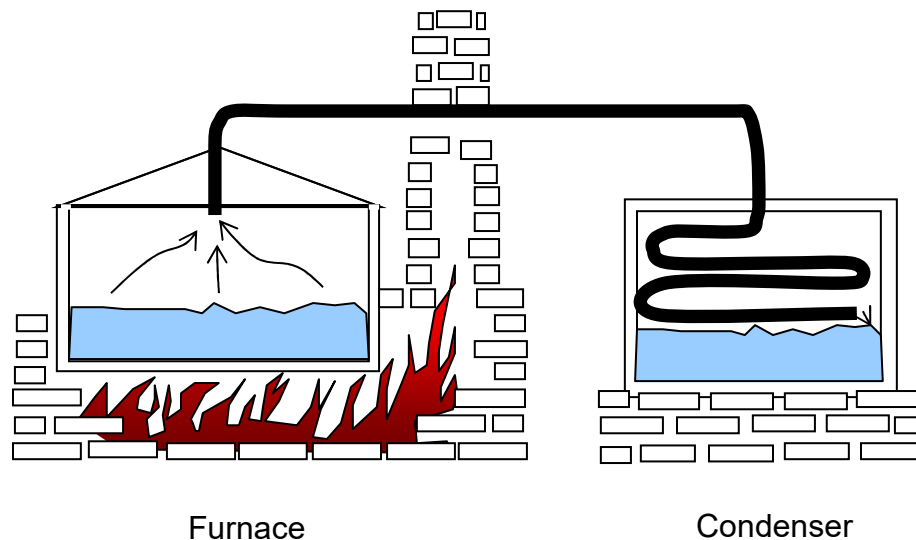


Figure 1 : Batch Still Distillation Process

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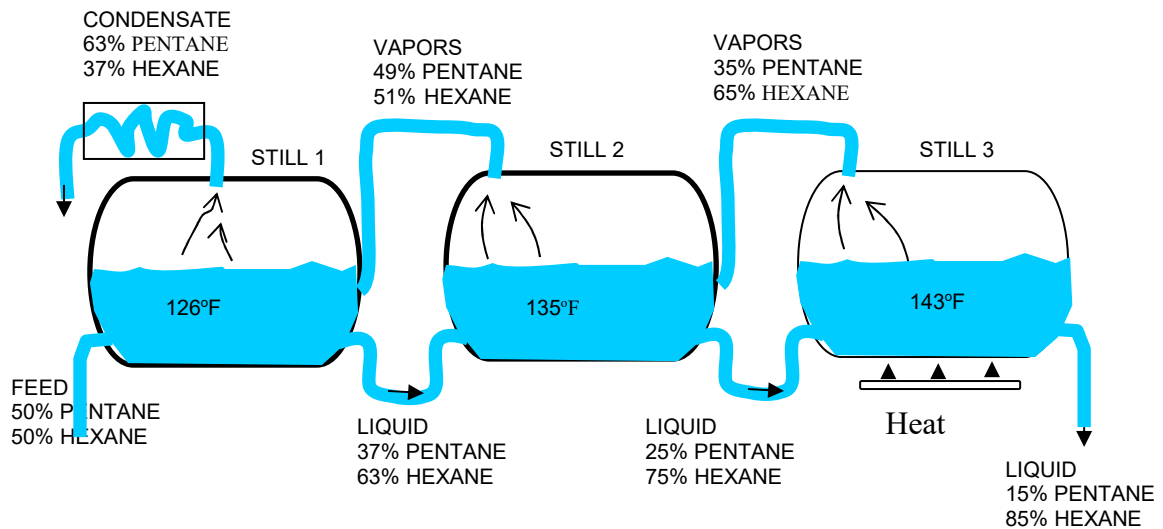


Figure 2 : Still Distillation in Series

Types of Distillation Processes

There are many types of distillation processes. Each type has its own characteristics and is designed to perform specific types of separations. These variations appear due to difficulty in separation when the physical properties of the components in a mixture are very close to one another, such as an azeotropic mixture.

One type of variation of the distillation processes is extractive distillation. In this type of process, an external solvent is added to the system to increase the separation. The external solvent changes the relative volatility between two 'close' components by extracting one of the components, forming a ternary mixture with different properties. The solvent is recycled into the system after the extracted component is separated from it.

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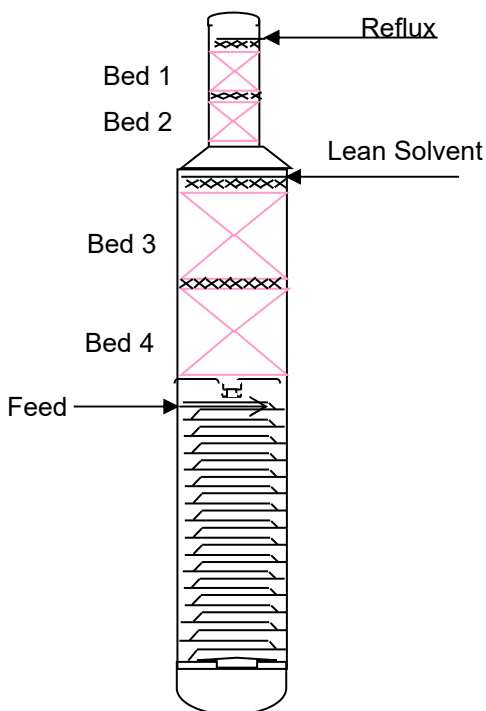


Figure 3 : Extractive Distillation Column

A distillation column may also have a catalyst bed and reaction occurring in it. This type of column is called a reactive distillation column. The targeted component reacts when it is in contact with the catalyst, thereby separated from the rest of the components in the mixture.

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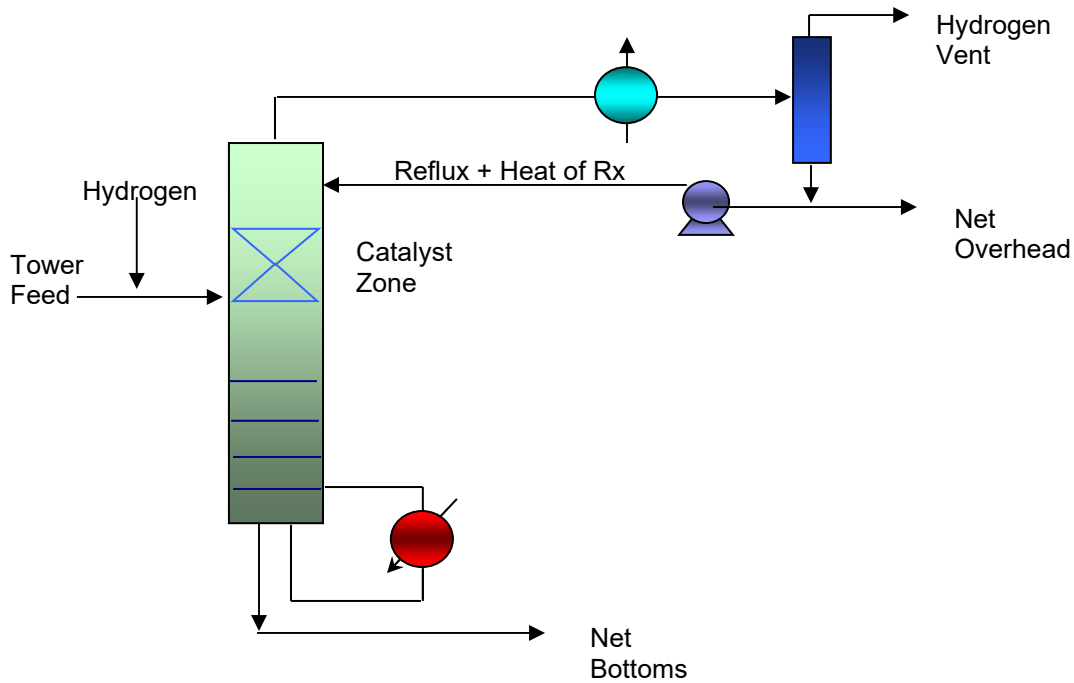


Figure 4 : Catalyst Distillation Column

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Mode of Operation

Distillation towers can be classified into two main categories, based on their mode of operation. The two classes are batch distillation and continuous distillation.

In batch distillation, the feed to the column is introduced batch-wise. The column is first charged with a 'batch' and then the distillation process is carried out. When the desired task is achieved, the next batch of feed is introduced. Batch distillation is usually preferred in the pharmaceutical industries and for the production of seasonal products.

On the other hand, continuous distillation handles a continuous feed stream. No interruption occurs during the operation of a continuous distillation column unless there is a problem with the column or surrounding unit operations. Continuous columns are capable of handling high throughputs. Besides, additional variations can be utilized in a continuous distillation column, such as multiple feed points and multiple product drawing points. Therefore, continuous columns are the more common of the two modes, especially in the petroleum and chemical industries.

Column Internals

Column internals are installed in distillation columns to provide better mass and heat transfers between the liquid and vapor phases in the column. These include trays, packings, distributors and redistributors, baffles and etc. They promote an intimate contact between both phases. The type of internals selected would determine the height and diameter of a column for a specified duty because different designs have various capacities and efficiencies. The two main types of column internals discussed in this guideline are trays and packing.

There are many types of trays or plates, such as sieve, bubble-cap and valve trays. Packing, on the other hand, can be categorized into random and structured packing. In random packing, rings and saddles are dumped into the column randomly while structured packing is stacked in a regular pattern in the column.

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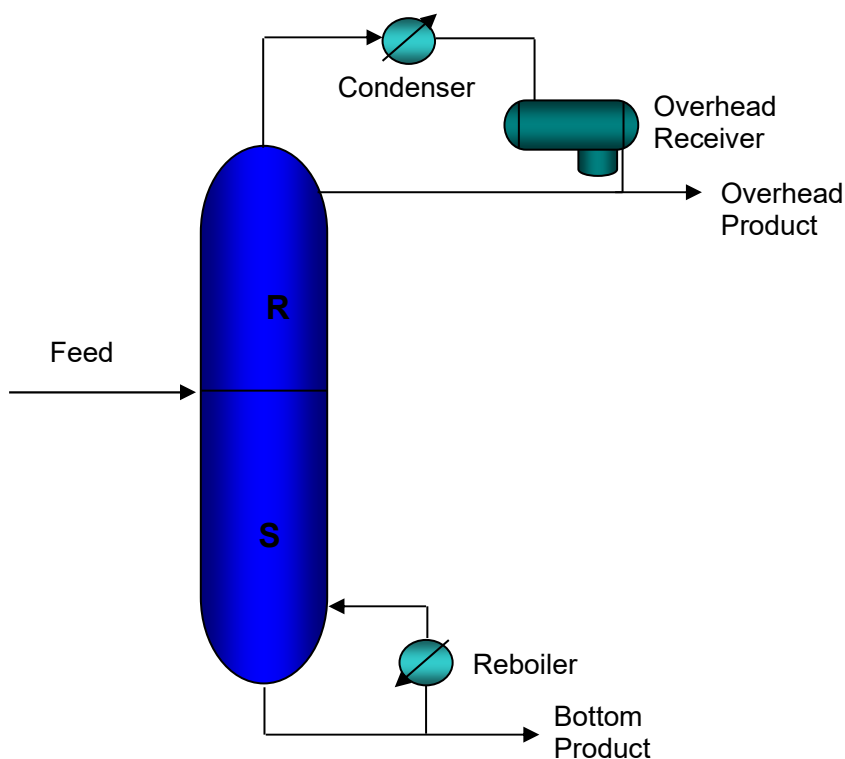


Figure 5 : Schematic Diagram of Distillation Column/ Fractionator

Figure 5 shows a schematic diagram of an example distillation column or fractionator. The feed enters the column as liquid, vapor or a mixture of vapor-liquid. The vapor phase that travels up the column is in contact with the liquid phase that travels down. Column distillation is divided two stages, there are rectifying stages and stripping stages.

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(A) Rectifying Stages

The process above the feed tray is known as rectification (where the vapor phase is continually enriched in the light components which will finally make up the overhead product). A liquid recycle condenses the less volatile components from rising vapor. To generate the liquid recycle, cooling is applied to condense a portion of the overhead vapor its name reflux.

(B) Stripping Stages

The process below the feed tray is known as stripping (as the heavier components are being stripped off and concentrated in the liquid phase to form the bottom product). At the top of the column, vapor enters the condenser where heat is removed. Some liquid is returned to the column as reflux to limit the loss of heavy components overhead.

At each separation stage (each tray or a theoretical stage in the packing), the vapor enters from the stage below at a higher temperature while the liquid stream enters from the stage above at a lower temperature. Heat and mass transfer occur such that the exiting streams (bubble point liquid and dew point vapor at the same temperature and pressure) are in equilibrium with each other.

(C) Condenser

The condenser above the column can be either a total or partial condenser. In a total condenser (Figure 6), all vapors leaving the top of the column is condensed to liquid so that the reflux stream and overhead product have the same composition.

In a partial condenser (Figure 7), only a portion of the vapor entering the condenser is condensed to liquid. In most cases, the condensed liquid is refluxed into the column and the overhead product drawn is in the vapor form. On the other hand, there are some cases where only part of the condensed liquid is refluxed. In these cases, there will be two overhead products, one a liquid with the same composition as the reflux stream while the other is a vapor product that is in equilibrium with the liquid reflux.

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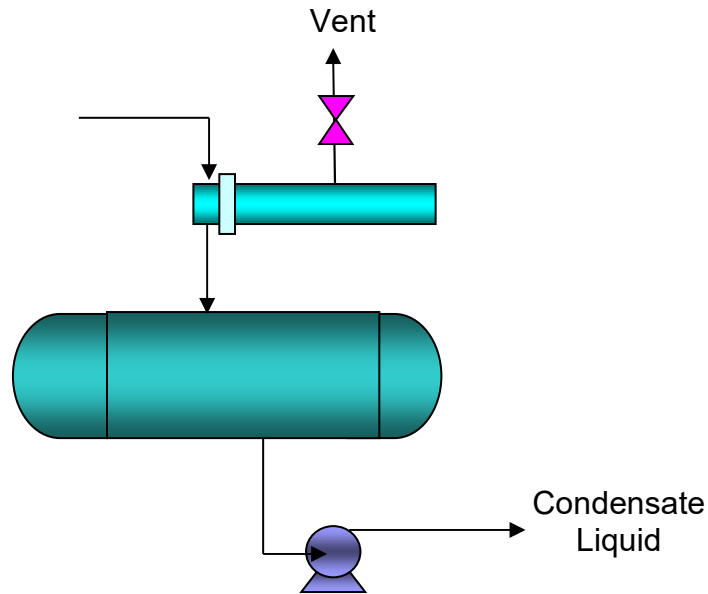


Figure 6 : Total Condenser

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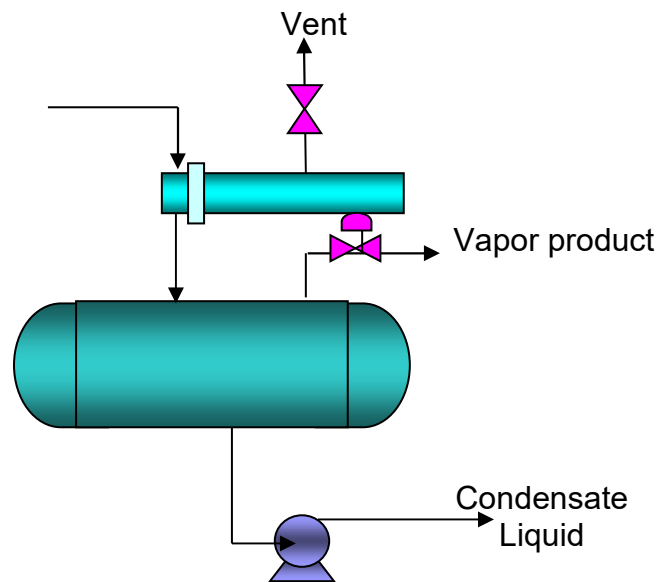


Figure 7 : Partial Condenser

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DEFINITIONS

Azeotrope- Is a mixture of two or more pure compounds (chemicals) in such a ratio that its composition cannot be changed by simple distillation. This is because when an azeotrope is boiled, the resulting vapor has the same ratio of constituents as the original mixture of liquids.

Bottoms – The stream of liquid product collected from the reboiler at the bottom of a distillation tower.

Bubble point – The temperature at constant pressure (or the pressure at constant temperature) at which the first vapor bubble forms when a liquid is heated (or decompressed).

Condenser- Is a heat exchanger which condenses a substance from its gaseous to its liquid state.

Dew point – The temperature at constant pressure (or the pressure at constant temperature) at which the first liquid droplet forms when a gas (vapor) is cooled (or compressed).

Distillate – The vapor from the top of a distillation column is usually condensed by a total or partial condenser. Part of the condensed fluid is recycled into the column (reflux) while the remaining fluid collected for further separation or as final product is known as distillate or overhead product.

Equation of state – A relation between the pressure, volume and temperature of a system, from which other thermodynamic properties may be derived. The relation employs any number of 'constants' specific to the system. For example, for a pure component, the constants may be generalized functions of critical temperature, critical pressure and acentric factor, while for a mixture, mixing rules (which may be dependent on composition or density), are also used.

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Heavy key – The heavier (less volatile) of the two key components. Heavy key is collected at the bottoms. All non-key components heavier than the heavy key are known as the heavy components.

Key component – A distillation column is assigned with two key components. The key components in the feed are the main components to be separated in that column. The volatility of the two key components must be in adjacent order when the volatilities of all the components in the feed are arranged in either ascending or descending order.

K-value – Vapor-liquid equilibrium constant or distribution coefficient. It is used in non-ideal (hydrocarbon) systems.

Light key – The lighter (more volatile) of the two key components. Light key is collected at the distillate. All non-key components lighter than the light key are known as the light components.

Reboiler – Is a heat exchanger typically used to provide heat to the bottom of industrial distillation columns. They boil the liquid from the bottom of a distillation column to generate vapors which are returned to the column to drive the distillation separation.

Reflux ratio – The ratio of the reflux stream to the distillate. The operating reflux ratio could affect the number of theoretical stages and the duties of reboiler and condenser.

Relative volatility – Relative volatility is defined as the ratio of the concentration of one component in the vapor over the concentration of that component in the liquid divided by the ratio of the concentration of a second component in the vapor over the concentration of that second component in the liquid. For an ideal system, relative volatility is the ratio of vapor pressures i.e. $\alpha = P_2/P_1$

Vapor-liquid equilibrium- Abbreviated as **VLE** by some, is a condition where a liquid and its vapor (gas phase) are in equilibrium with each other, a condition or state where the rate of evaporation (liquid changing to vapor) equals the rate of condensation (vapor changing to liquid) on a molecular level such that there is no net (overall) vapor-liquid interconversion

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Vapor pressure – The pressure exerted by the vapor phase that is in equilibrium with the liquid phase in a closed system. For moderate temperature ranges, the vapor pressure at a given temperature can be estimated using the Antoine equation.

Weir loading – The normalized liquid flow rate leaving a tray pass divided by the length of the outlet weir of the same pass.

Open area - The ratio of the hole area divided by the bubbling area.

Constriction factor at the bottom downcomer - The ratio of the outlet weir length over the tower diameter.

The downcomer clearance - The distance between the bottom edge of the downcomer apron and the tray deck

NOMENCLATURE

B	Bottom product rate, moles/unit time
<i>b</i>	Bottoms product flow rate, ft ³ /min
C	Coefficient, ft/hr
CFS	Vapor loading, ft ³ /s
D	Distillate product rate, moles/unit time
D _T	Tower diameter, ft
<i>d</i>	Distillate flow rate, ft ³ /min
F	Feed rate, moles/unit time
<i>f_i</i>	Fugacity of component <i>i</i>
H	Tower height, ft
H _{BP}	Enthalpy of bubble point feed stream, Btu/hr
H _{VF}	Enthalpy of vaporized feed stream, Btu/hr
<i>K</i>	Vapor-liquid equilibrium constant
L _o	Reflux liquid, moles/unit time
L _R	Liquid molar rate in the rectification section
L _S	Liquid molar rate in the stripping section
<i>N</i>	Number of theoretical stages

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N_m	Minimum number of theoretical stages
P	Total system pressure, psi
P^*	Vapor pressure, psi
Q	Reboiler duty, Btu/hr
Q_c	Condenser duty, Btu/hr
q	Thermal condition of feed
V_1	Vapor rate at overhead column, moles/unit time
V_{calc}	Calculated vapor rate, moles/unit time
V_{corr}	Corrected vapor rate, moles/unit time
V_{max}	Maximum volumetric flow rate, ft ³ /hr
v_{max}	Maximum velocity, ft/hr
R	Reflux ratio
R_m	Minimum reflux ratio
S_F	Separation factor,
T	Temperature, °F
x	Mole fraction in the liquid phase
X_B	Bottom liquid rate, moles/unit time
x_d	Mole fraction in the distillate
X_{Di}	Mole fraction of component i in the distillate
X_D	Distillate liquid rate, moles/unit time
x_f	Mole fraction in the feed
X_{Fi}	Mole fraction of component i in the feed
x_w	Mole fraction in the bottoms
y	Mole fraction in the vapor phase

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Greek letters

α	relative volatility
γ	activity coefficient
φ	vapor phase fugacity coefficient
β	volatility factor
ρ	density, lb/ft ³

Superscripts

L	liquid phase
V	vapor phase
b	exponent

Subscripts

avg	average
bottom	bottom section of column
HHK	heavy component
HK	heavy key
<i>i</i>	component <i>i</i>
<i>j</i>	component <i>j</i>
LK	light key
LLK	light component
top	top section of column

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