

Distillation

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an introduction

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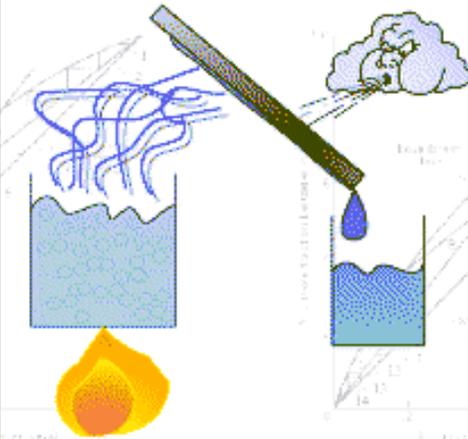
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Distillation is defined as:

a process in which a liquid or vapour mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat.



Distillation is based on the fact that the vapour of a boiling mixture will be richer in the components that have lower boiling points.

Therefore, when this vapour is cooled and condensed, the condensate will contain more volatile components. At the same time, the original mixture will contain more of the less volatile material.

Distillation columns are designed to achieve this separation efficiently.

Although many people have a fair idea what "distillation" means, the important aspects that seem to be missed from the manufacturing point of view are that:

- ➔ distillation is the most common separation technique
- ➔ it consumes enormous amounts of energy, both in terms of cooling and heating requirements
- ➔ it can contribute to more than 50% of plant operating costs

The best way to reduce operating costs of existing units, is to improve their efficiency and operation via process optimisation and control. To achieve this improvement, a thorough understanding of distillation principles and how distillation systems are designed is essential.

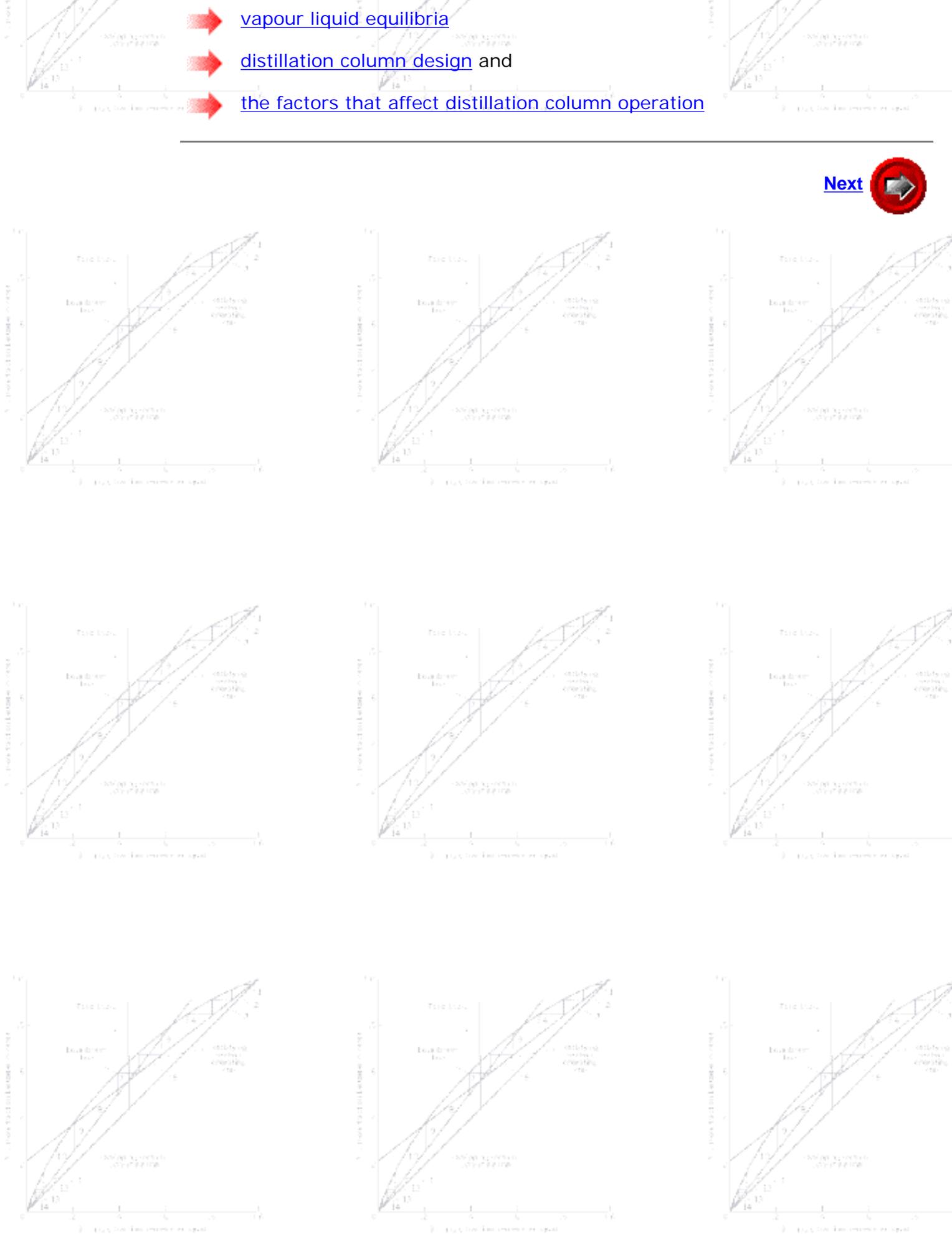
The purpose of this set of notes is to expose you to the terminology used in distillation practice and to give a very basic introduction to:

- ➔ [types of columns](#)
- ➔ [basic distillation equipment and operation](#)
- ➔ [column internals](#)
- ➔ [reboilers](#)
- ➔ [distillation principles](#)

→ vapour liquid equilibria

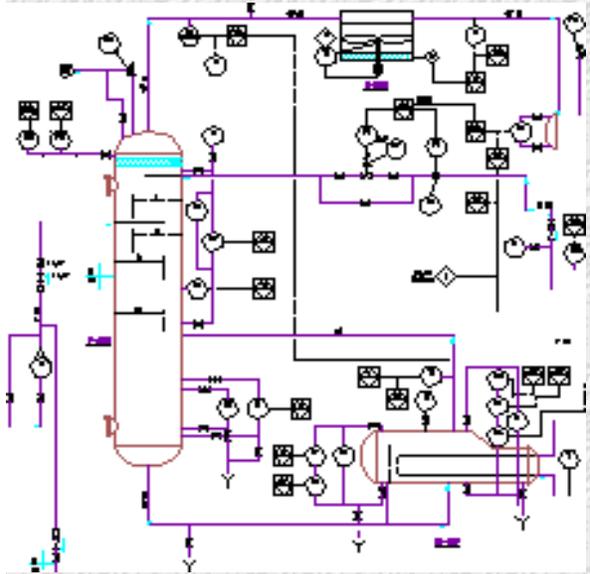
→ distillation column design and

→ the factors that affect distillation column operation



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An excellent document [Distillation - An Introduction](#) is highly recommended reading.

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Alkylation

Amination

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[Esterification](#)

Etherification

Hydrolysis

Isomerization

Oligomerization

[Transesterification](#)

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Distillation Column Internals / Distillation Design Software

Major Producers of distillation column internals are listed below for your convenience. Many have free software that is available by request.

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[Jaeger Products, Inc.](#)

[Sulzer Chemtech](#)

[Amistco](#)

[Raschig](#)

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TYPES OF DISTILLATION COLUMNS

There are many types of distillation columns, each designed to perform specific types of separations, and each design differs in terms of complexity.

Batch and Continuous Columns

One way of classifying distillation column type is to look at how they are operated. Thus we have:

- ➔ **batch** and
- ➔ **continuous** columns.

Batch Columns

In batch operation, the feed to the column is introduced batch-wise. That is, the column is charged with a 'batch' and then the distillation process is carried out. When the desired task is achieved, a next batch of feed is introduced.

Continuous Columns

In contrast, continuous columns process a continuous feed stream. No interruptions occur unless there is a problem with the column or surrounding process units. They are capable of handling high throughputs and are the most common of the two types. We shall concentrate only on this class of columns.

Types of Continuous Columns

Continuous columns can be further classified according to:

the nature of the feed that they are processing,

- ➔ **binary** column - feed contains only two components
- ➔ **multi-component** column - feed contains more than two components

the number of product streams they have

- ➔ **multi-product** column - column has more than two product streams

where the extra feed exits when it is used to help with the separation,

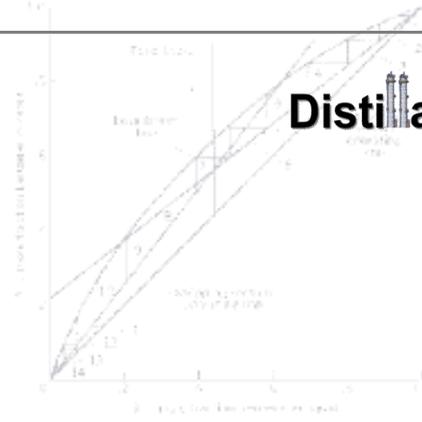
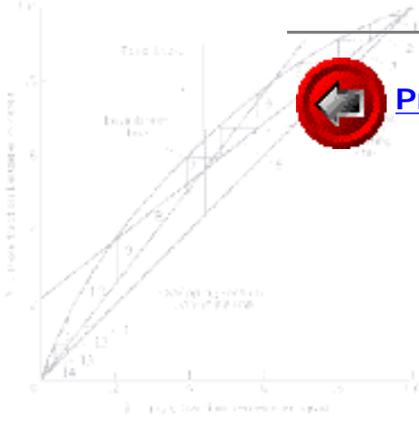
- ➔ **extractive** distillation - where the extra feed appears in the bottom product stream

→ **azeotropic** distillation - where the extra feed appears at the top product stream

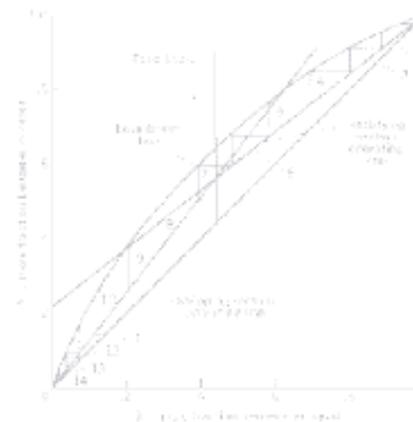
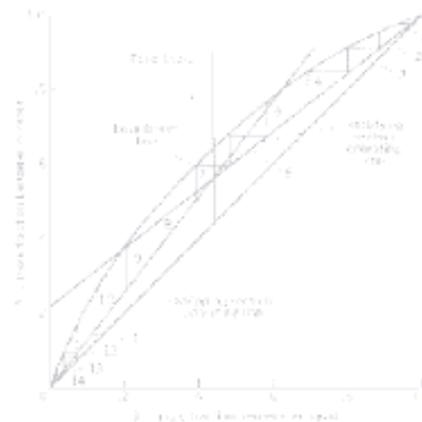
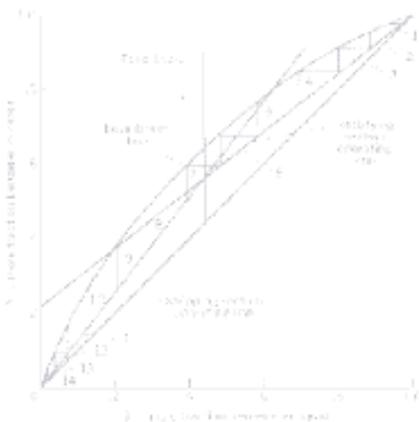
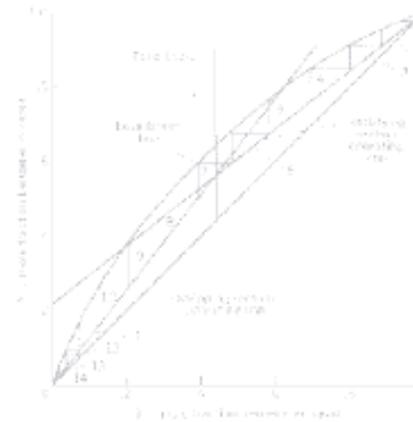
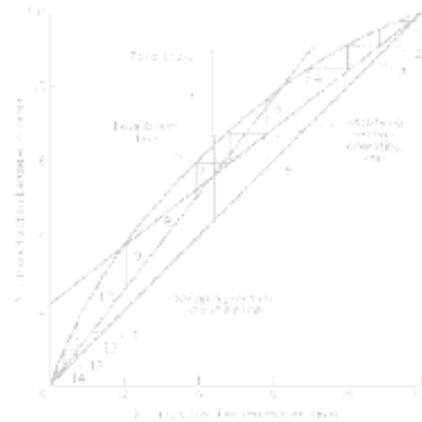
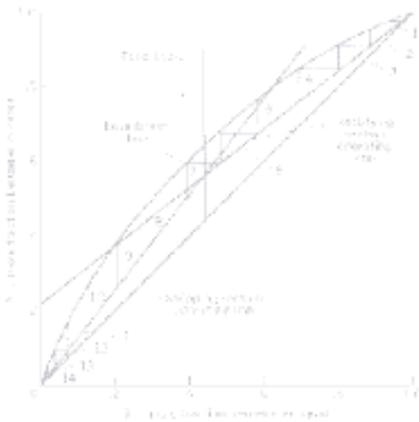
the type of column internals

→ **tray** column - where trays of various designs are used to hold up the liquid to provide better contact between vapour and liquid, hence better separation

→ **packed** column - where instead of trays, 'packings' are used to enhance contact between vapour and liquid



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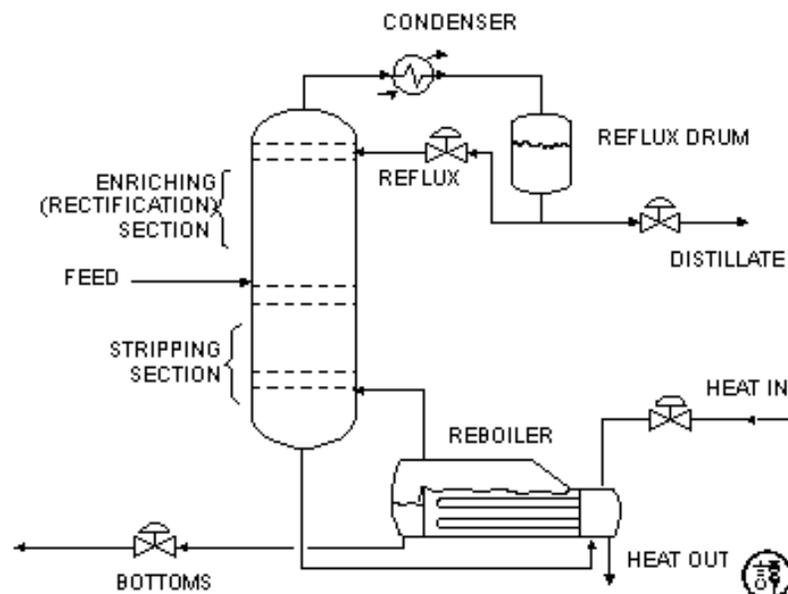
BASIC DISTILLATION EQUIPMENT AND OPERATION

Main Components of Distillation Columns

Distillation columns are made up of several components, each of which is used either to transfer heat energy or enhance material transfer. A typical distillation contains several major components:

- ➔ a vertical **shell** where the separation of liquid components is carried out
- ➔ column internals such as **trays/plates** and/or **packings** which are used to enhance component separations
- ➔ a **reboiler** to provide the necessary vaporisation for the distillation process
- ➔ a **condenser** to cool and condense the vapour leaving the top of the column
- ➔ a **reflux drum** to hold the condensed vapour from the top of the column so that liquid (**reflux**) can be recycled back to the column

The vertical shell houses the column internals and together with the condenser and reboiler, constitute a distillation column. A schematic of a typical distillation unit with a single feed and two product streams is shown below:

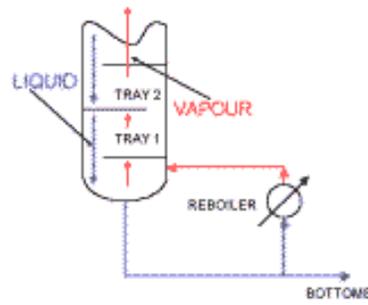


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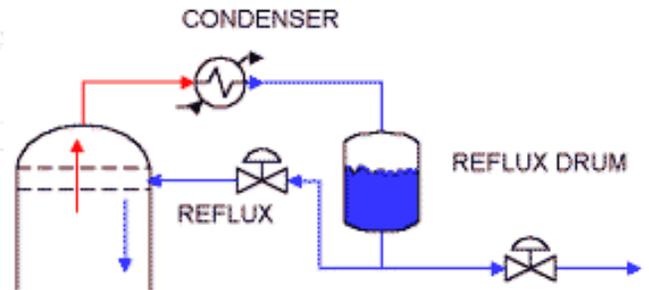
The liquid mixture that is to be processed is known as the **feed** and this is introduced usually somewhere near the middle of the column to a **tray** known as the **feed tray**. The feed tray divides the column into a top (**enriching** or **rectification**) section and a bottom (**stripping**) section. The feed flows down the column where it is collected at the bottom in the **reboiler**.



Heat is supplied to the reboiler to generate vapour. The source of heat input can be any suitable fluid, although in most chemical plants this is normally steam. In refineries, the heating source may be the output streams of other columns. The vapour raised in the reboiler is re-introduced into the unit at the bottom of the column. The liquid removed from the reboiler is known as the **bottoms product** or simply,

bottoms.

The vapour moves up the column, and as it exits the top of the unit, it is cooled by a **condenser**. The condensed liquid is stored in a holding vessel known as the **reflux drum**. Some of this liquid is recycled back to the top of the column and this is called the **reflux**. The condensed liquid that is removed from the system is known as the **distillate** or **top product**.



Thus, there are **internal flows** of vapour and liquid within the column as well as **external flows** of feeds and product streams, into and out of the column.



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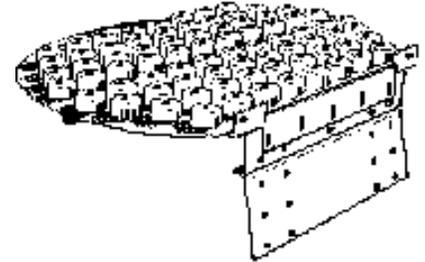


Trays and Plates

The terms "trays" and "plates" are used interchangeably. There are many types of tray designs, but the most common ones are :

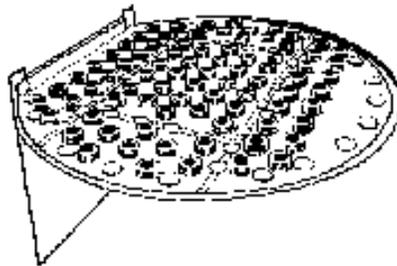
Bubble cap trays

A bubble cap tray has riser or chimney fitted over each hole, and a cap that covers the riser. The cap is mounted so that there is a space between riser and cap to allow the passage of vapour. Vapour rises through the chimney and is directed downward by the cap, finally discharging through slots in the cap, and finally bubbling through the liquid on the tray.



Valve trays

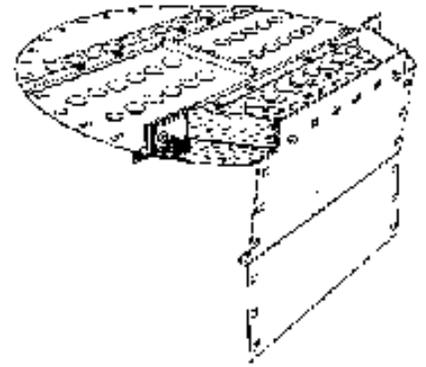
In valve trays, perforations are covered by liftable caps. Vapour flows lifts the caps, thus self creating a flow area for the passage of vapour. The lifting cap directs the vapour to flow horizontally into the liquid, thus providing better mixing than is possible in sieve trays.



Valve trays (photos courtesy of Paul Phillips)

Sieve trays

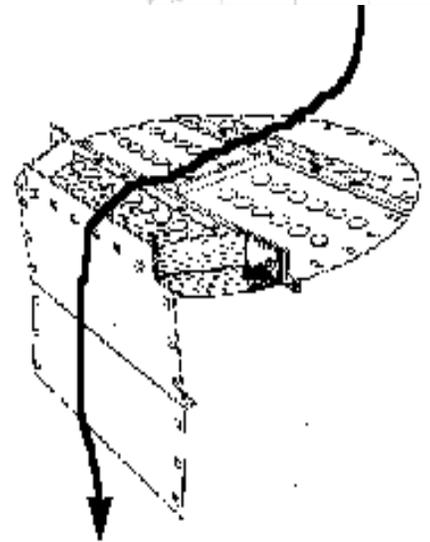
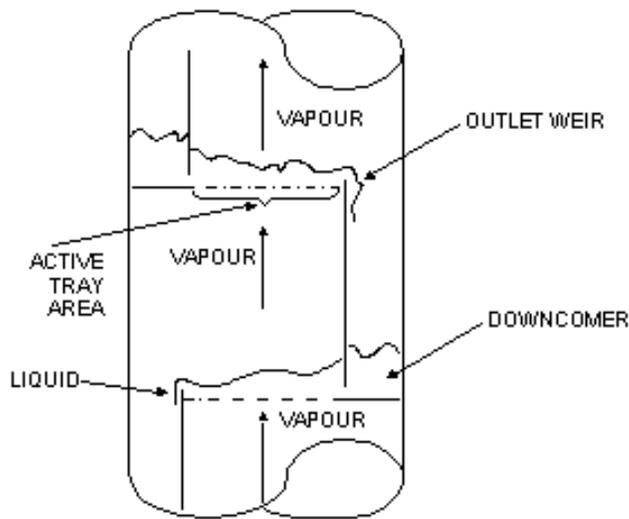
Sieve trays are simply metal plates with holes in them. Vapour passes straight upward through the liquid on the plate. The arrangement, number and size of the holes are design parameters.



Because of their efficiency, wide operating range, ease of maintenance and cost factors, sieve and valve trays have replaced the once highly thought of bubble cap trays in many applications.

Liquid and Vapour Flows in a Tray Column

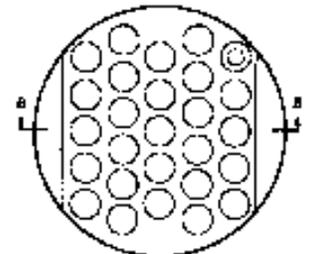
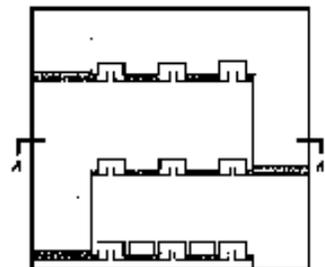
The next few figures show the direction of vapour and liquid flow across a tray, and across a column.



Each tray has 2 conduits, one on each side, called '**downcomers**'. Liquid falls through the downcomers by gravity from one tray to the one below it. The flow across each plate is shown in the above diagram on the right.

A **weir** on the tray ensures that there is always some liquid (**holdup**) on the tray and is designed such that the the holdup is at a suitable height, e.g. such that the bubble caps are covered by liquid.

Being lighter, vapour flows up the column and is forced to pass through the liquid, via the openings on each tray. The area allowed for the passage of vapour on each tray is called the **active tray area**.



The picture on the left is a photograph of a section of a pilot scale column equipped with bubble capped trays. The tops of the 4 bubble caps on the tray can just be seen. The down-comer in this case is a pipe, and is shown on the right. The frothing of the liquid on



the active tray area is due to both passage of vapour from the tray below as well as boiling.

As the hotter vapour passes through the liquid on the tray above, it transfers heat to the liquid. In doing so, some of the vapour condenses adding to the liquid on the tray. The condensate, however, is richer in the less volatile components than is in the vapour. Additionally, because of the heat input from the vapour, the liquid on the tray boils, generating more vapour. This vapour, which moves up to the next tray in the column, is richer in the more volatile components. This continuous contacting between vapour and liquid occurs on each tray in the column and brings about the separation between low boiling point components and those with higher boiling points.

Tray Designs

A tray essentially acts as a mini-column, each accomplishing a fraction of the separation task. From this we can deduce that the more trays there are, the better the degree of separation and that overall separation efficiency will depend significantly on the design of the tray. Trays are designed to maximise vapour-liquid contact by considering the

- ➔ liquid distribution and
- ➔ vapour distribution

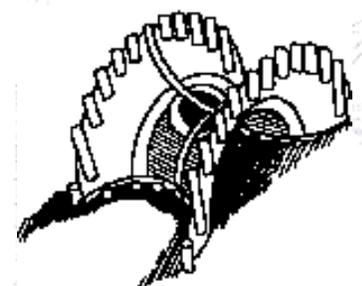
on the tray. This is because better vapour-liquid contact means better separation at each tray, translating to better column performance. Less trays will be required to achieve the same degree of separation. Attendant benefits include less energy usage and lower construction costs.



Liquid distributors - Gravity (left), Spray (right)
(photos courtesy of Paul Phillips)

Packings

There is a clear trend to improve separations by supplementing the use of trays by additions of packings. Packings are passive devices that are designed to increase the interfacial area for vapour-liquid contact. The following pictures show 3 different types of packings.



These strangely shaped pieces are supposed to impart good vapour-liquid contact when a particular type is placed together in numbers, without causing excessive pressure-drop across a packed section. This is important because a high pressure drop would mean that more energy is required to drive the vapour up the distillation column.



Structured packing (photo courtesy of Paul Phillips)

Packings versus Trays

A tray column that is facing throughput problems may be de-bottlenecked by replacing a section of trays with packings. This is because:

- ➔ packings provide extra inter-facial area for liquid-vapour contact
- ➔ efficiency of separation is increased for the same column height
- ➔ packed columns are shorter than trayed columns

Packed columns are called **continuous-contact columns** while trayed columns are called **staged-contact columns** because of the manner in which vapour and liquid are contacted.



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COLUMN REBOILERS

There are a number of designs of reboilers. It is beyond the scope of this set of introductory notes to delve into their design principles. However, they can be regarded as heat-exchangers that are required to transfer enough energy to bring the liquid at the bottom of the column to boiling point. The following are examples of typical reboiler types.

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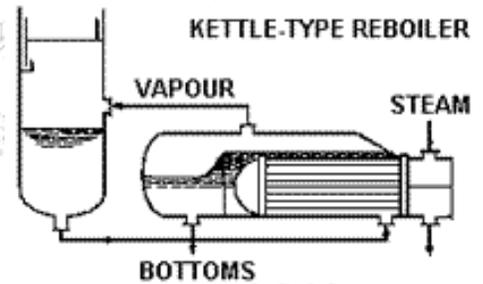
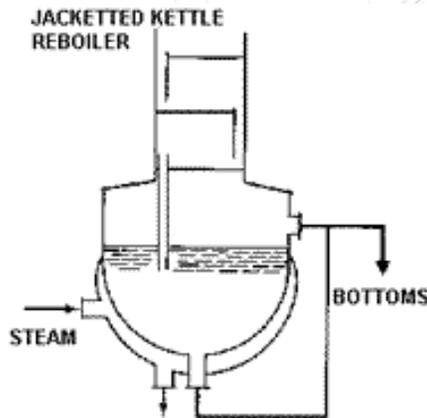
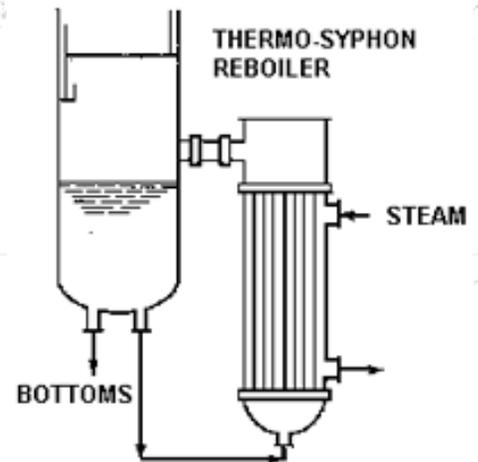
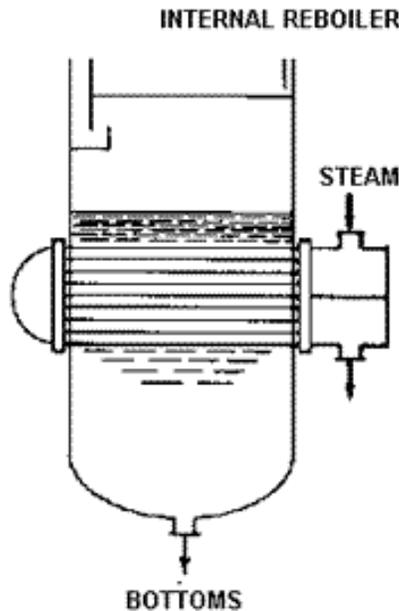


Photo courtesy of Brian Kennedy





Tube bundle
(photo courtesy of Paul Phillips)



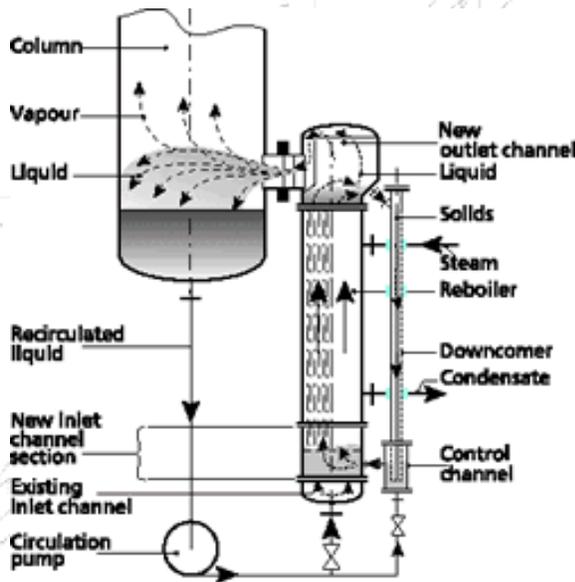
Tube bundle
(photo courtesy of Paul Phillips)



Tubesheet
(photo courtesy of Paul Phillips)



Fitting a Shell & Tube Heat Exchanger
(photo courtesy of Paul Phillips)



A novel development in reboiler design is the self-cleaning shell-and-tube heat exchangers by Klarex Technology for applications where heat exchange surfaces are prone to fouling by the process fluid. Particles are introduced into the process stream and these produce a scouring action on the heat exchange surfaces. An example is shown in the diagram on the left. [[Click on it to find out more](#)]



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DISTILLATION PRINCIPLES

Separation of components from a liquid mixture via distillation depends on the differences in boiling points of the individual components. Also, depending on the concentrations of the components present, the liquid mixture will have different boiling point characteristics. Therefore, distillation processes depends on the **vapour pressure** characteristics of liquid mixtures.

Vapour Pressure and Boiling

The vapour pressure of a liquid at a particular temperature is the **equilibrium pressure** exerted by molecules leaving and entering the liquid surface. Here are some important points regarding vapour pressure:

- ➔ energy input raises vapour pressure
- ➔ vapour pressure is related to boiling
- ➔ a liquid is said to 'boil' when its vapour pressure equals the surrounding pressure
- ➔ the ease with which a liquid boils depends on its **volatility**
- ➔ liquids with high vapour pressures (volatile liquids) will boil at lower temperatures
- ➔ the vapour pressure and hence the boiling point of a liquid mixture depends on the relative amounts of the components in the mixture
- ➔ distillation occurs because of the differences in the volatility of the components in the liquid mixture

The Boiling Point Diagram

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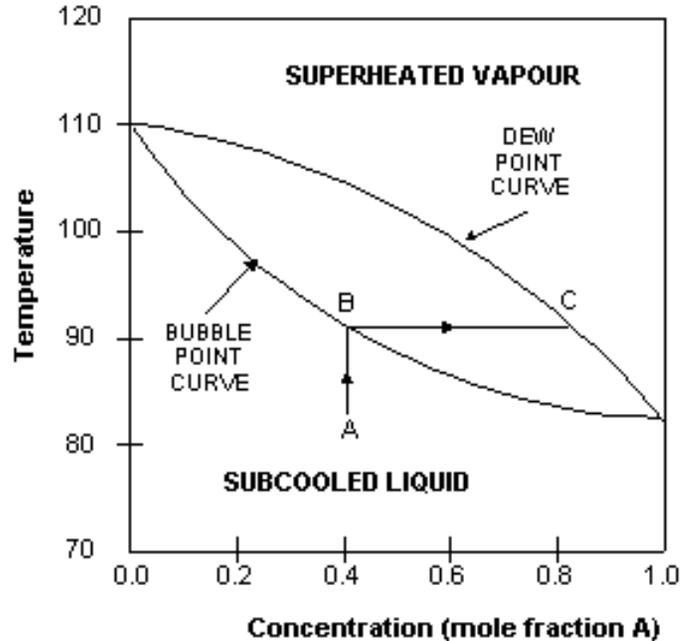
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The **boiling point diagram** shows how the equilibrium compositions of the components in a liquid mixture vary with temperature at a fixed pressure. Consider an example of a liquid mixture containing 2 components (A and B) - a **binary** mixture. This has the following boiling point diagram.



The boiling point of A is that at which the mole fraction of A is 1. The boiling point of B is that at which the mole fraction of A is 0. In this example, A is the more volatile component and therefore has a lower boiling point than B. The upper curve in the diagram is called the **dew-point curve** while the lower one is called the **bubble-point curve**.

The **dew-point** is the temperature at which the saturated vapour starts to condense.

The **bubble-point** is the temperature at which the liquid starts to boil.

The region above the dew-point curve shows the equilibrium composition of the **superheated** vapour while the region below the bubble-point curve shows the equilibrium composition of the **subcooled** liquid.

For example, when a subcooled liquid with mole fraction of $A=0.4$ (point A) is heated, its concentration remains constant until it reaches the bubble-point (point B), when it starts to boil. The vapours evolved during the boiling has the equilibrium composition given by point C, approximately 0.8 mole fraction A. This is approximately 50% richer in A than the original liquid.

This difference between liquid and vapour compositions is the basis for distillation operations.

Relative Volatility

Relative volatility is a measure of the differences in volatility between 2 components, and hence their boiling points. It indicates how easy or difficult a particular separation will be. The relative volatility of component 'i' with respect to component 'j' is defined as

$$\alpha_{ij} = \frac{\left(\frac{y_i}{x_i}\right)}{\left(\frac{y_j}{x_j}\right)}$$

y_i = mole fraction of component 'i' in the vapour
 x_i = mole fraction of component 'i' in the liquid

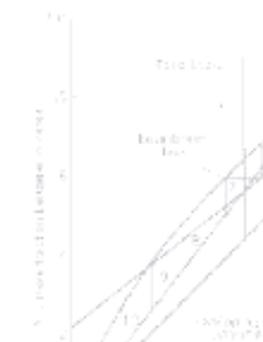
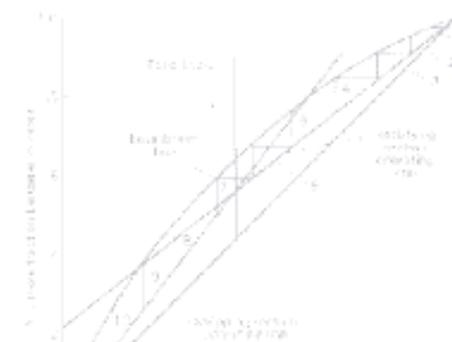
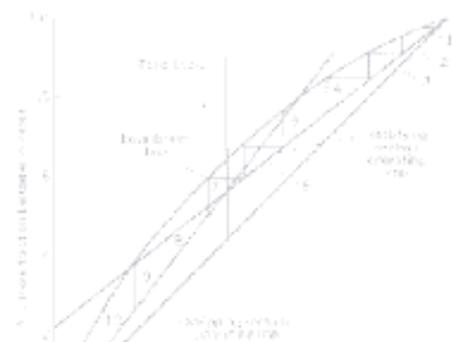
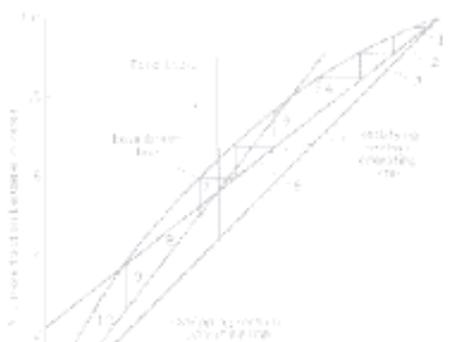
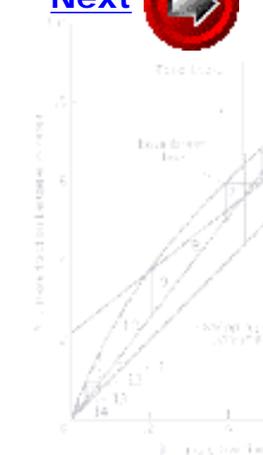
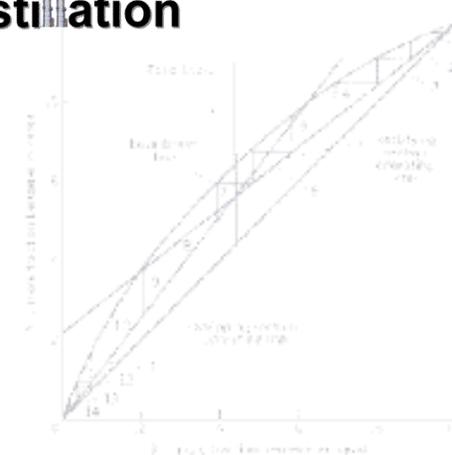
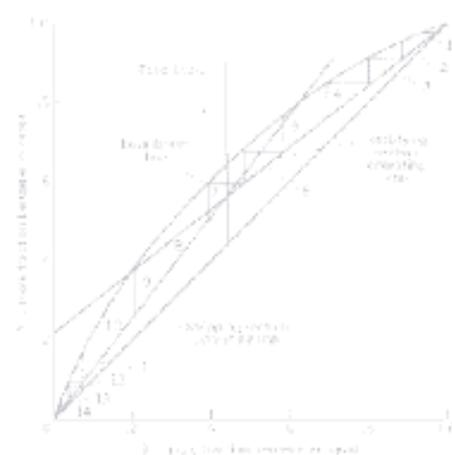
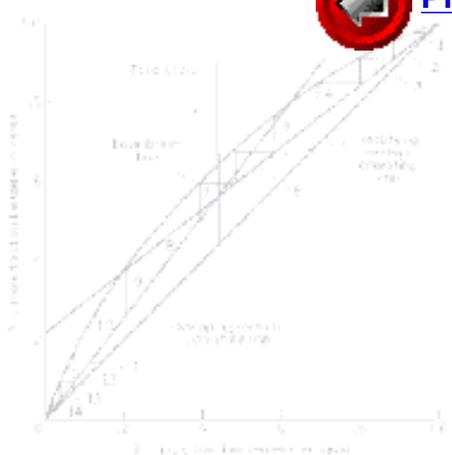
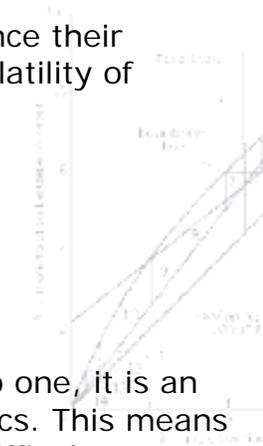
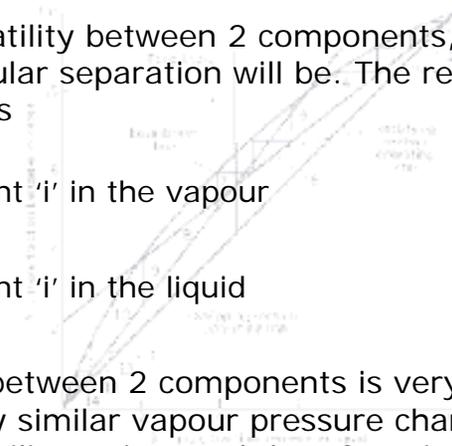
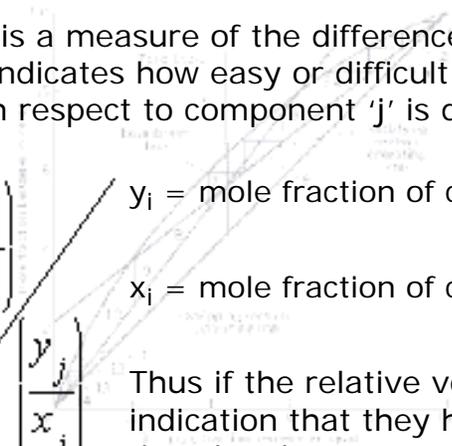
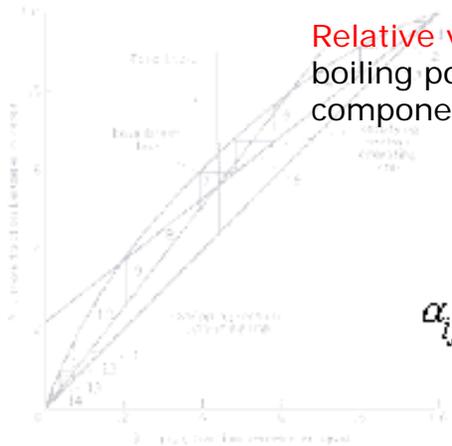
Thus if the relative volatility between 2 components is very close to one, it is an indication that they have very similar vapour pressure characteristics. This means that they have very similar boiling points and therefore, it will be difficult to separate the two components via distillation.



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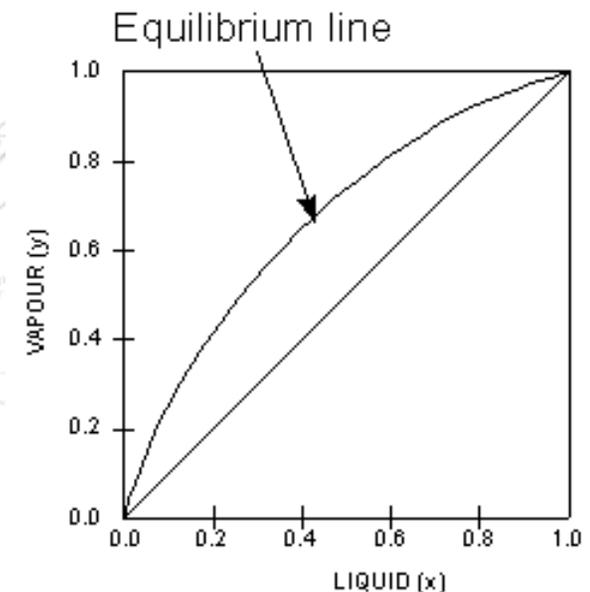
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VAPOUR LIQUID EQUILIBRIA

Distillation columns are designed based on the boiling point properties of the components in the mixtures being separated. Thus the sizes, particularly the height, of distillation columns are determined by the vapour liquid equilibrium (VLE) data for the mixtures.

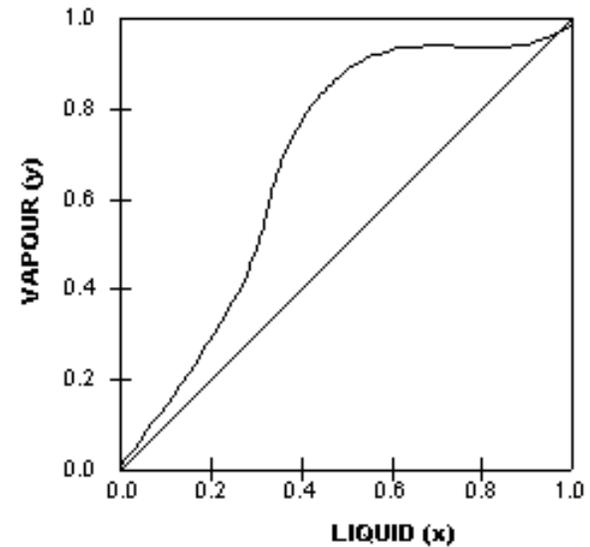
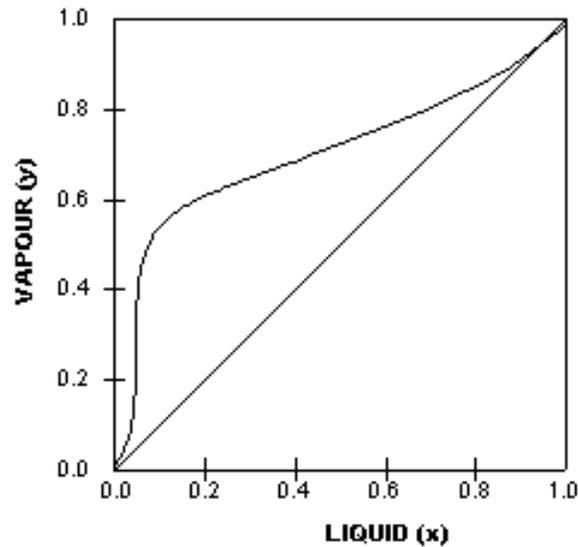
Vapour-Liquid-Equilibrium (VLE) Curves

Constant pressure VLE data is obtained from [boiling point diagrams](#). VLE data of binary mixtures is often presented as a plot, as shown in the figure on the right. The VLE plot expresses the bubble-point and the dew-point of a binary mixture at constant pressure. The curved line is called the **equilibrium line** and describes the compositions of the liquid and vapour in equilibrium at some fixed pressure.

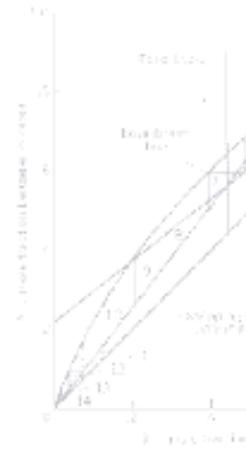
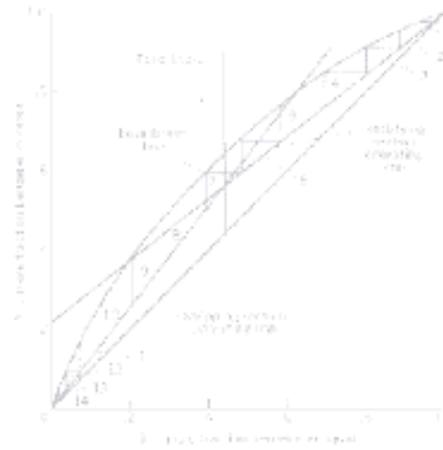
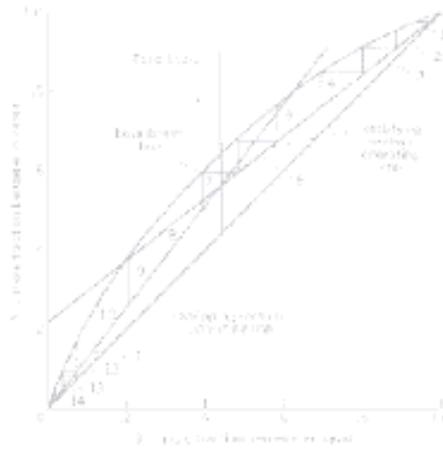
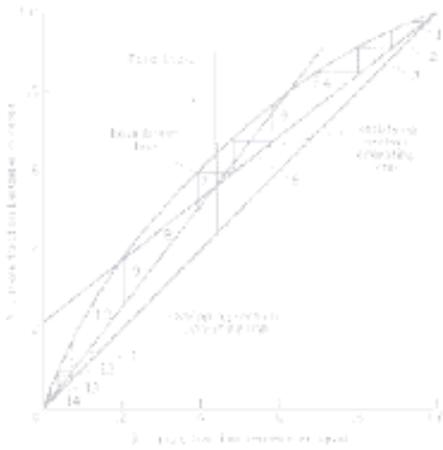


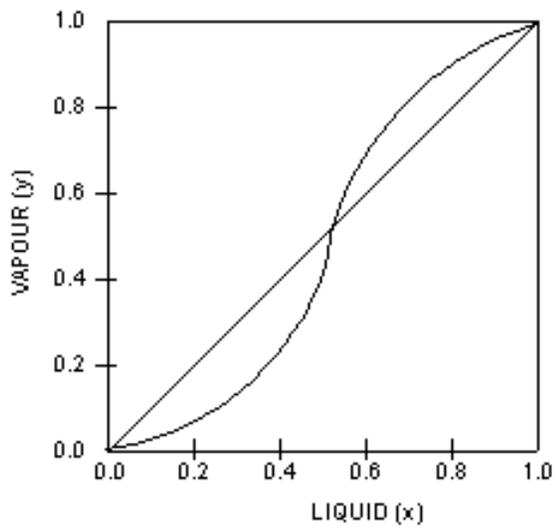
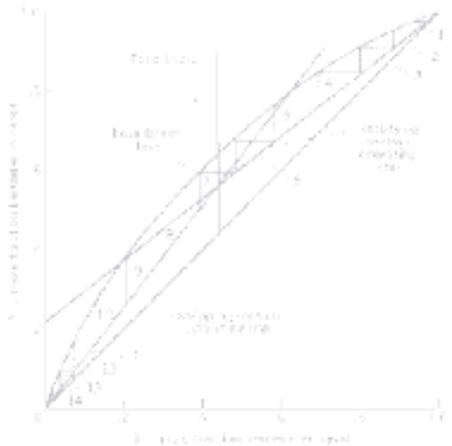
This particular VLE plot shows a binary mixture that has a uniform vapour-liquid equilibrium that is relatively easy to separate. The next two VLE plots below on the other hand, shows non-ideal systems which will present more difficult separations. We can tell from the shapes of the curves and this will be explained further later on.

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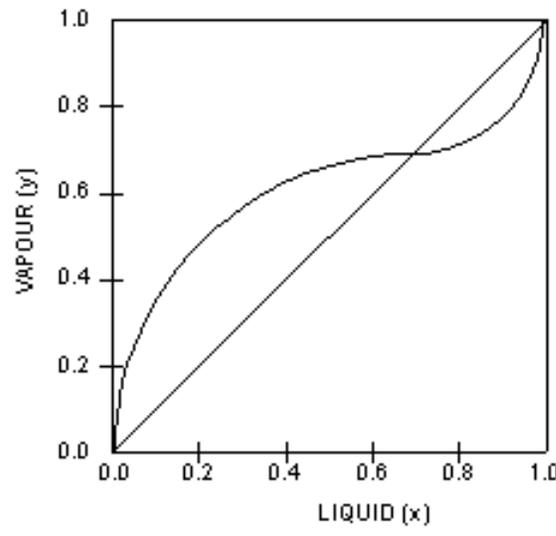


The most intriguing VLE curves are generated by azeotropic systems. **An azeotrope is a liquid mixture which when vapourised, produces the same composition as the liquid.** The two VLE plots below, show two different azeotropic systems, one with a minimum boiling point and one with a maximum boiling point. In both plots, the equilibrium curves cross the diagonal lines, and this are **azeotropic points** where the azeotropes occur. In other words azeotropic systems give rise to VLE plots where the equilibrium curves crosses the diagonals.

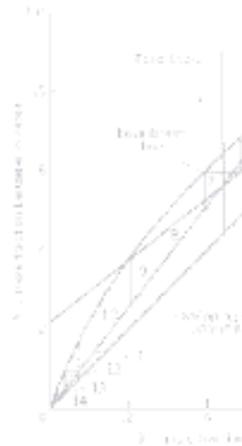




Maximum Boiling Point



Minimum Boiling Point

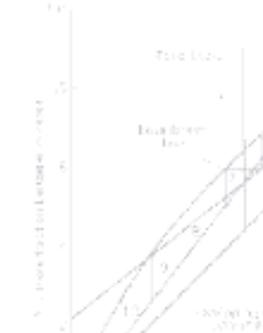
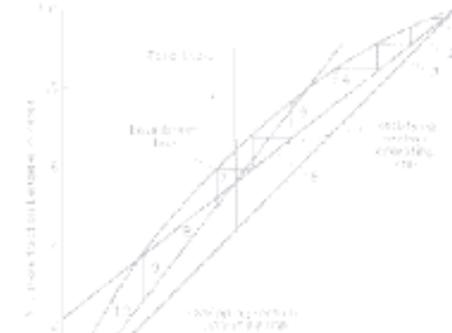
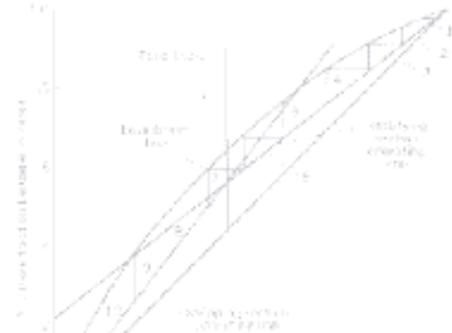
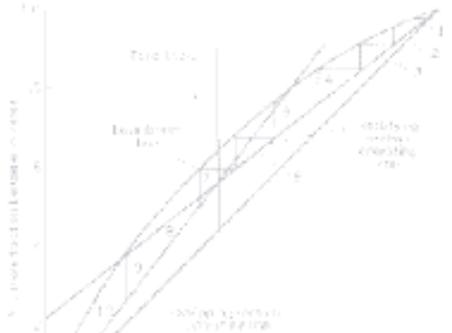


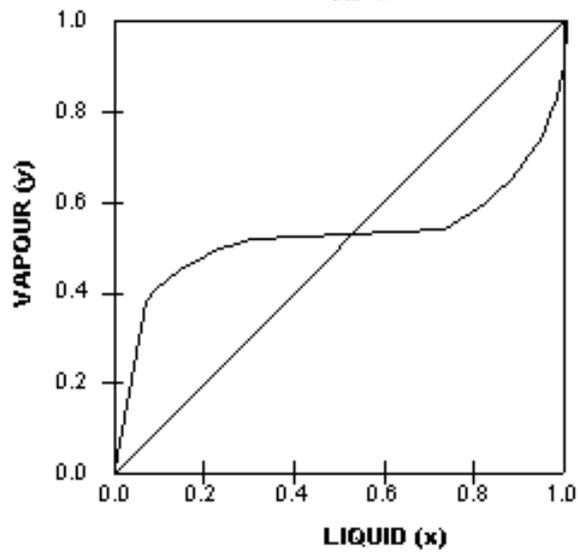
Note the shapes of the respective equilibrium lines in relation to the diagonal lines that bisect the VLE plots.

Both plots are however, obtained from homogenous azeotropic systems. An azeotrope that contains one liquid phase in contact with vapour is called a **homogenous azeotrope**. A homogenous azeotrope cannot be separated by conventional distillation. However, **vacumn distillation** may be used as the lower pressures can shift the azeotropic point. Alternatively, an additional substance may added to shift the azeotropic point to a more 'favourable' position.

➔ When this additional component appears in appreciable amounts at the top of the column, the operation is called **azeotropic distillation**.

➔ When the additional component appears mostly at the bottom of the column, the operation is called **extractive distillation**





The VLE curve on the left is also generated by an azeotropic system, in this case a **heterogenous azeotrope**. Heterogenous azeotropes can be identified by the 'flat' portion on the equilibrium diagram.

They may be separated in 2 distillation columns since these substances usually form two liquid phases with widely differing compositions. The phases may be separated using settling tanks under appropriate conditions.

Next, we will look at how VLE plots/data are used to design distillation columns.



Distillation



Distillation

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DISTILLATION COLUMN DESIGN

As mentioned, distillation columns are designed using [VLE data](#) for the mixtures to be separated. The vapour-liquid equilibrium characteristics (indicated by the shape of the equilibrium curve) of the mixture will determine the number of stages, and hence the number of trays, required for the separation. This is illustrated clearly by applying the **McCabe-Thiele** method to design a binary column.

McCABE-THIELE DESIGN METHOD

The McCabe-Thiele approach is a graphical one, and uses the VLE plot to determine the theoretical number of stages required to effect the separation of a binary mixture. It assumes **constant molar overflow** and this implies that:

- molal heats of vaporisation of the components are roughly the same
- heat effects (heats of solution, heat losses to and from column, etc.) are negligible
- for every mole of vapour condensed, 1 mole of liquid is vaporised

The design procedure is simple. Given the VLE diagram of the binary mixture, operating lines are drawn first.

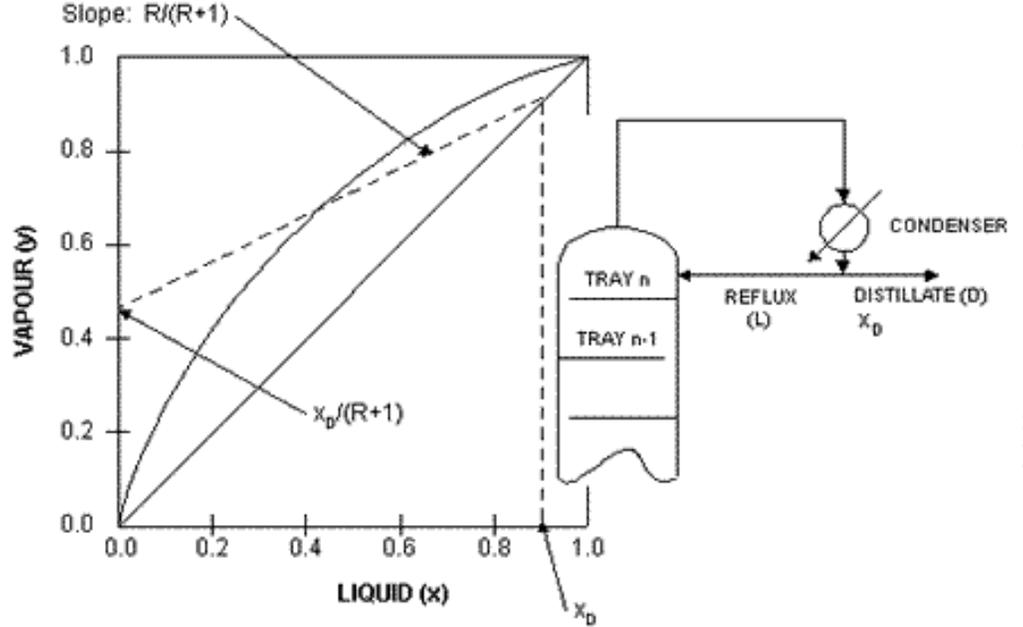
- Operating lines define the mass balance relationships between the liquid and vapour phases in the column.
- There is one operating line for the bottom (stripping) section of the column, and one for the top (rectification or enriching) section of the column.
- Use of the constant molar overflow assumption also ensures the operating lines are straight lines.

Operating Line for the Rectification Section

The operating line for the rectification section is constructed as follows. First the desired top product composition is located on the VLE diagram, and a vertical line produced until it intersects the diagonal line that splits the VLE plot in half. A line with slope $R/(R+1)$ is then drawn from this intersection point as shown in the diagram below.

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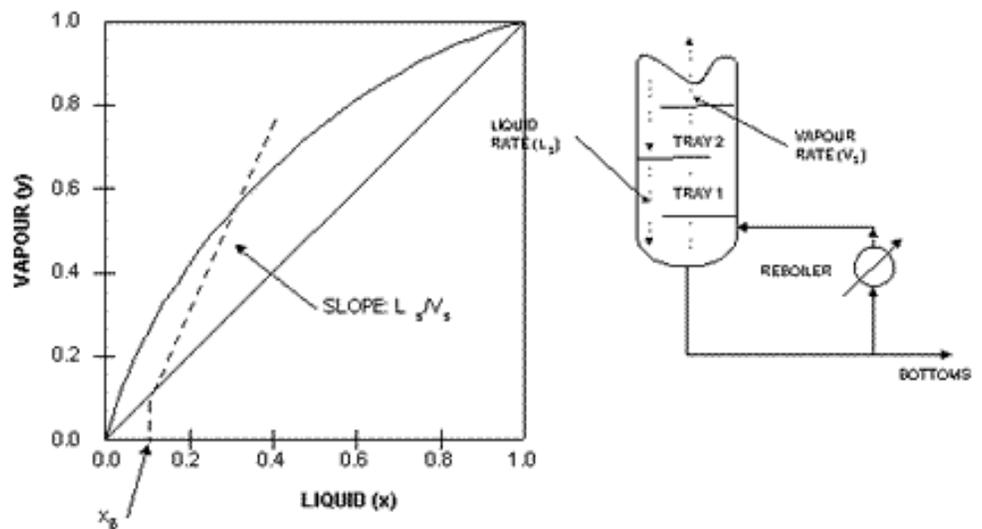




R is the ratio of reflux flow (L) to distillate flow (D) and is called the **reflux ratio** and is a measure of how much of the material going up the top of the column is returned back to the column as reflux.

Operating Line for the Stripping Section

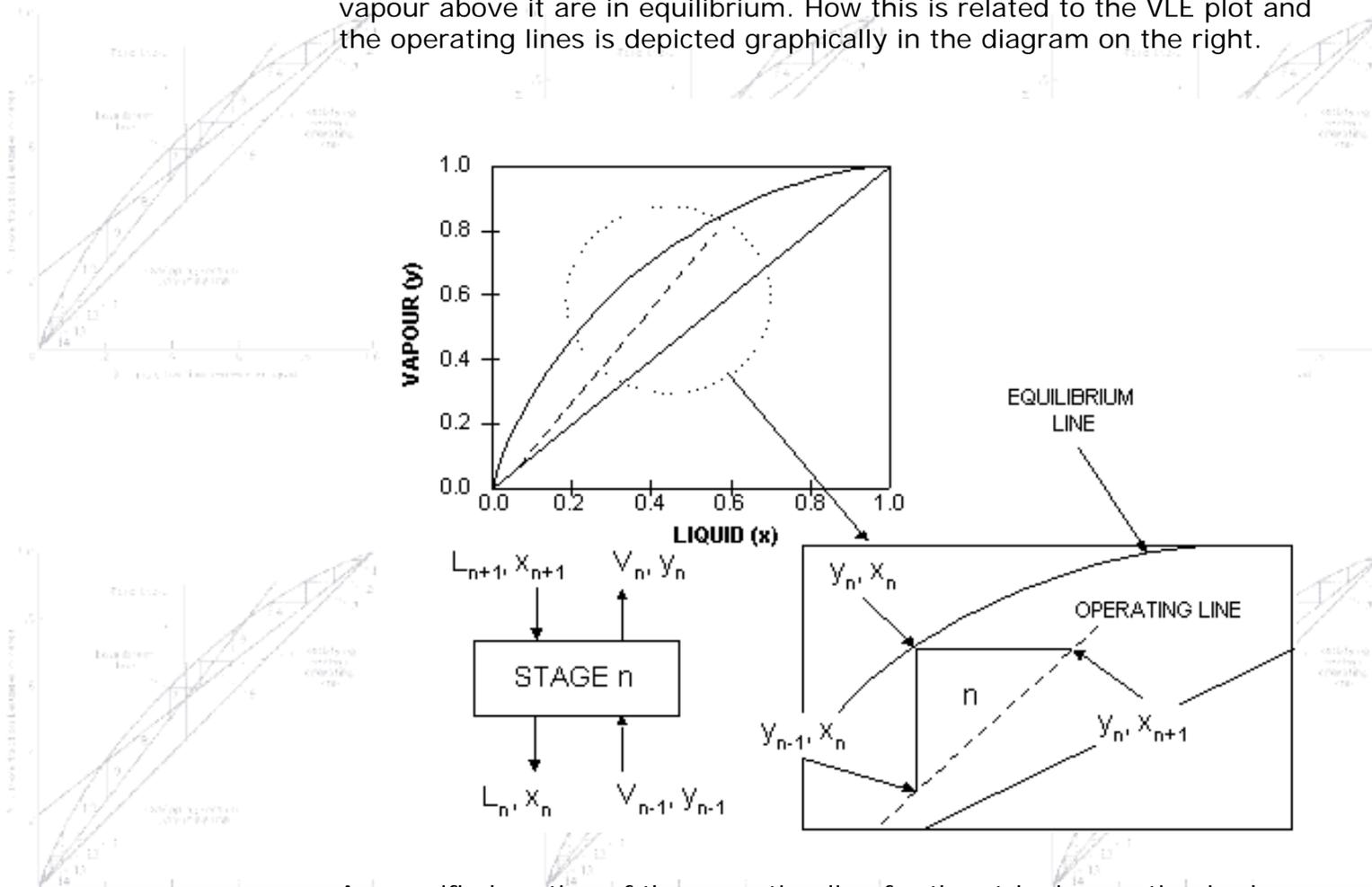
The operating line for the stripping section is constructed in a similar manner. However, the starting point is the desired bottom product composition. A vertical line is drawn from this point to the diagonal line, and a line of slope L_s/V_s is drawn as illustrated in the diagram below.



L_s is the liquid rate down the stripping section of the column, while V_s is the vapour rate up the stripping section of the column. Thus the slope of the operating line for the stripping section is a ratio between the liquid and vapour flows in that part of the column.

Equilibrium and Operating Lines

The McCabe-Thiele method assumes that the liquid on a tray and the vapour above it are in equilibrium. How this is related to the VLE plot and the operating lines is depicted graphically in the diagram on the right.

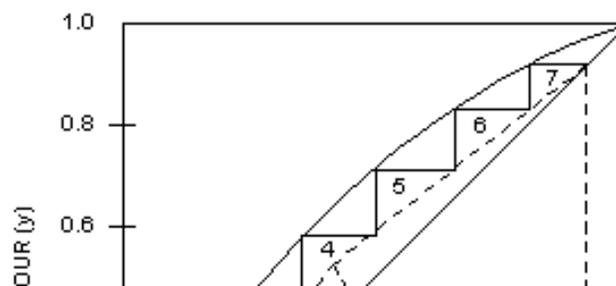


A magnified section of the operating line for the stripping section is shown in relation to the corresponding n 'th stage in the column. L 's are the liquid flows while V 's are the vapour flows. x and y denote liquid and vapour compositions and the subscripts denote the **origin** of the flows or compositions. That is ' $n-1$ ' will mean **from the stage below stage ' n '** while ' $n+1$ ' will mean **from the stage above stage ' n '**. The liquid in stage ' n ' and the vapour above it are in equilibrium, therefore, x_n and y_n

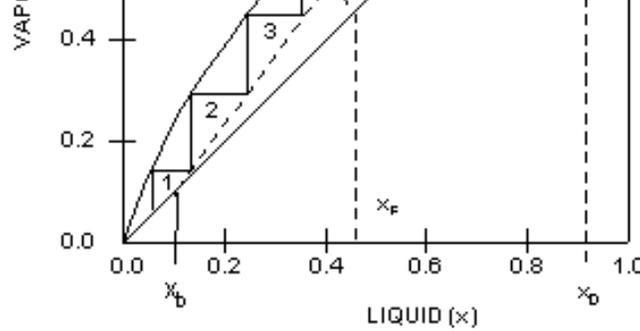
lie on the equilibrium line. Since the vapour is carried to the tray above without changing composition, this is depicted as a horizontal line on the VLE plot. Its intersection with the operating line will give the composition of the liquid on tray ' $n+1$ ' as the operating line defines the material balance on the trays. The composition of the vapour above the ' $n+1$ ' tray is obtained from the intersection of the vertical line from this point to the equilibrium line.

Number of Stages and Trays

Doing the graphical construction repeatedly will give rise to a number of 'corner' sections, and each section will be equivalent to a stage of the distillation. This is the basis of sizing distillation columns using the McCabe-Thiele graphical design methodology as shown in the following example.



Given the operating lines for both stripping and rectification sections, the graphical construction described above was applied. This particular example shows that **7 theoretical stages** are required to



achieve the desired separation. The required number of trays (as opposed to stages) is one less than the number of stages since the graphical construction includes the contribution of the reboiler in carrying out the separation.

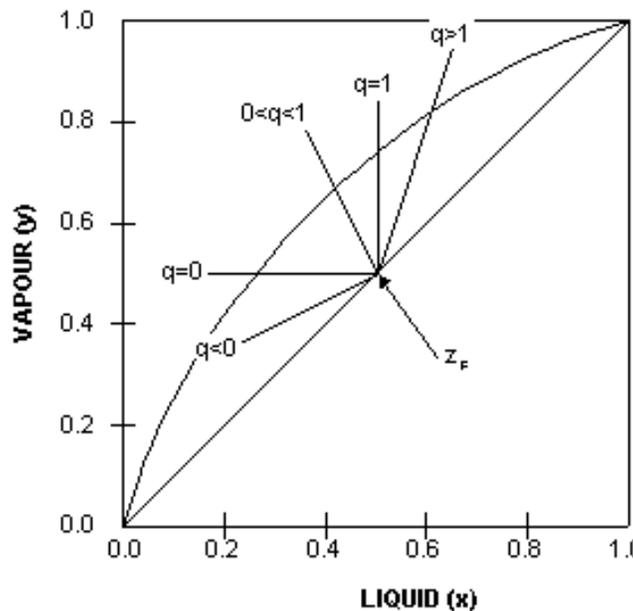
The actual number of trays required is given by the formula:

$$\text{(number of theoretical trays)} / \text{(tray efficiency)}$$

Typical values for tray efficiency ranges from 0.5 to 0.7 and depends on a number of factors, such as the [type of trays](#) being used, and internal liquid and vapour flow conditions. Sometimes, additional trays are added (up to 10%) to accommodate the possibility that the column may be under-designed.

The Feed Line (q-line)

The diagram above also shows that the binary feed should be introduced at the 4'th stage. However, if the feed composition is such that it does not coincide with the intersection of the operating lines, this means that the feed is not a saturated liquid. The condition of the feed can be deduced by the slope of the **feed line** or **q-line**. The q-line is that drawn between the intersection of the operating lines, and where the feed composition lies on the diagonal line.



Depending on the state of the feed, the feed lines will have different slopes. For example,

- $q = 0$ (saturated vapour)
- $q = 1$ (saturated liquid)
- $0 < q < 1$ (mix of liquid and vapour)
- $q > 1$ (subcooled liquid)
- $q < 0$ (superheated vapour)

The q-lines for the various feed conditions are shown in the diagram on the left.

Using Operating Lines and the Feed Line in McCabe-Thiele Design

If we have information about the condition of the feed mixture, then we can construct the q-line and use it in the McCabe-Thiele design. However, excluding the equilibrium line, only two other pairs of lines can be used in the McCabe-Thiele procedure. These are:

- feed-line and rectification section operating line
- feed-line and stripping section operating line
- stripping and rectification operating lines

This is because these pairs of lines determine the third.

[see Flash tutorial on [Distillation Basics](#) written by Jon Lee]

OVERALL COLUMN DESIGN

Determining the number of stages required for the desired degree of separation and the location of the feed tray is merely the first steps in producing an overall distillation column design. Other things that need to be considered are tray spacings; column diameter; internal configurations; heating and cooling duties. All of these can lead to conflicting design parameters. Thus, distillation column design is often an iterative procedure. If the conflicts are not resolved at the design stage, then the column will not perform well in practice. The next set of notes will discuss the factors that can affect distillation column performance.



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EFFECTS OF THE NUMBER OF TRAYS OR STAGES

Here we will expand on the design of columns by looking briefly at the effects of

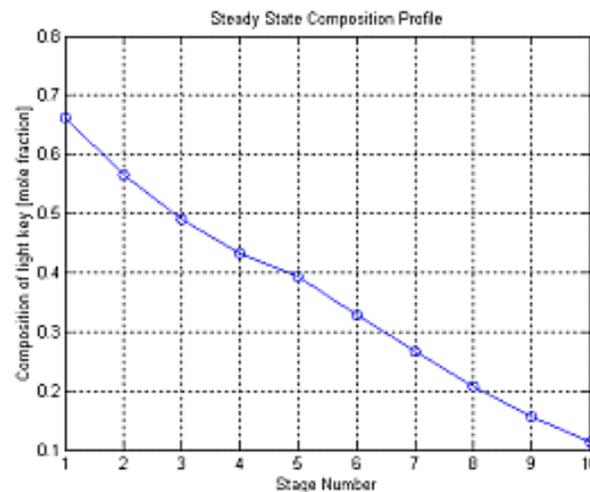
- the number of trays, and
- the position of the feed tray, and

on the performances of distillation columns.

Effects of the Number of Trays

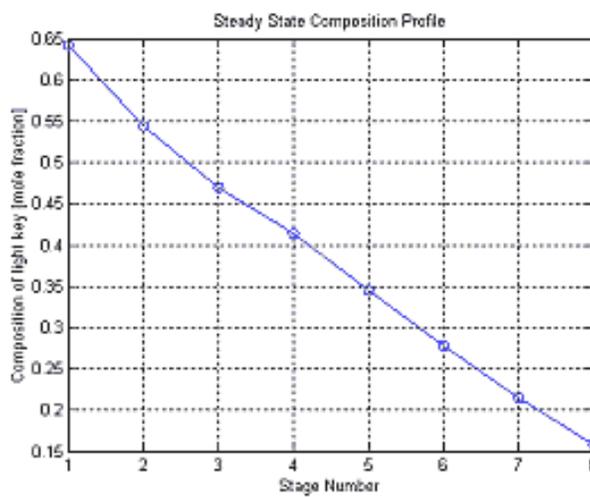
It can be deduced from the previous section on [distillation column design](#) that the number of trays will influence the degree of separation. This is illustrated by the following example.

Consider as a base case, a 10 stage column. The feed is a binary mixture that has a composition of 0.5 mole fraction in terms of the more volatile component, and introduced at stage 5. The steady-state terminal compositions of about 0.65 at the top (stage 1) and 0.1 at the bottom (stage 10) are shown below:



Composition Profile: 10 stages, feed at stage 5

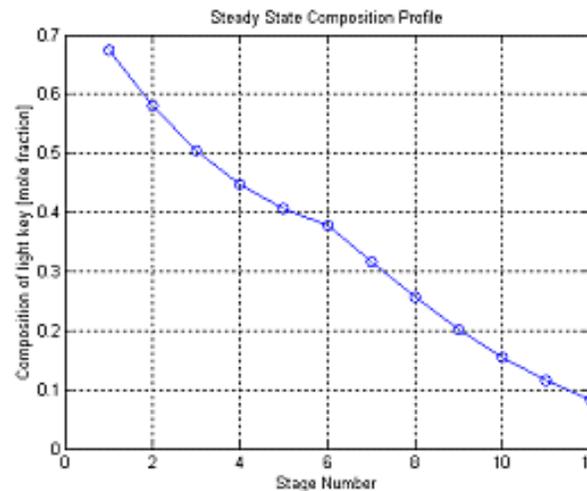
Suppose we **decrease** the number of stages to 8, and keep the feed at the middle stage, i.e. stage 4. The resulting composition profile is:



Composition Profile: 8 stages, feed at stage 4

We can see that the top composition has decreased while the bottom composition has increased. That is, the separation is poorer.

Now, if we **increase** the number of stages to 12, and again introduce the feed at mid-column, i.e. stage 6, the composition profile we get is:



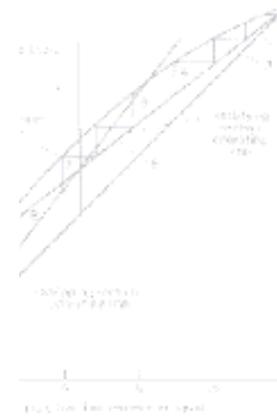
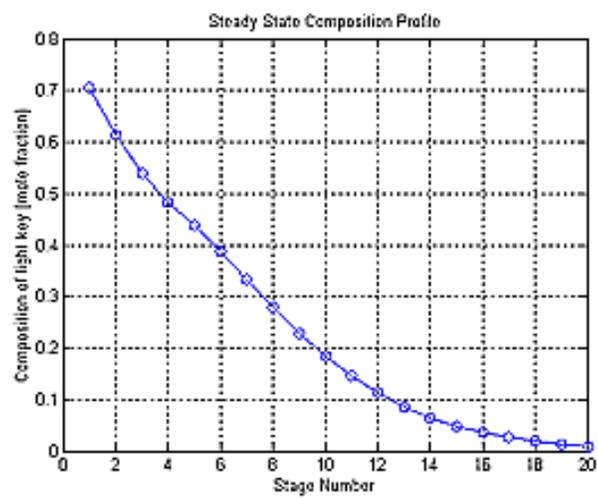
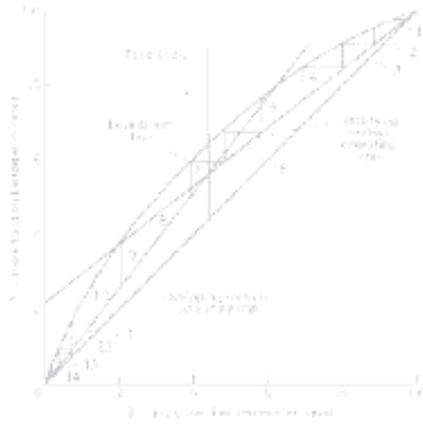
Composition Profile: 12 stages, feed at stage 6

Again, the composition has changed. This time the distillate is much richer in the more volatile component, while the bottoms has less, indicating better separation.

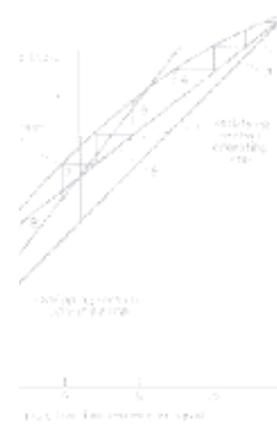
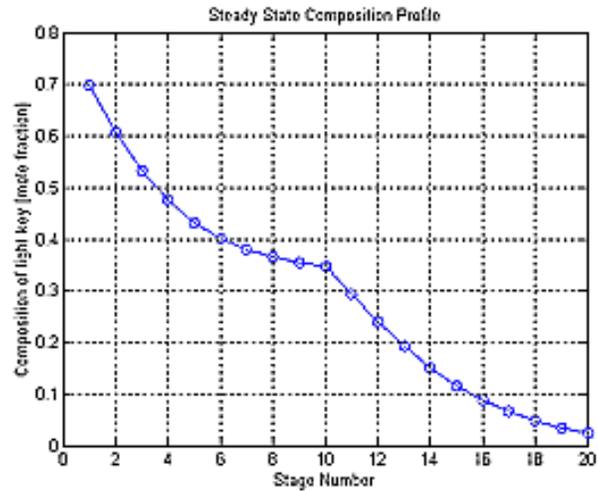
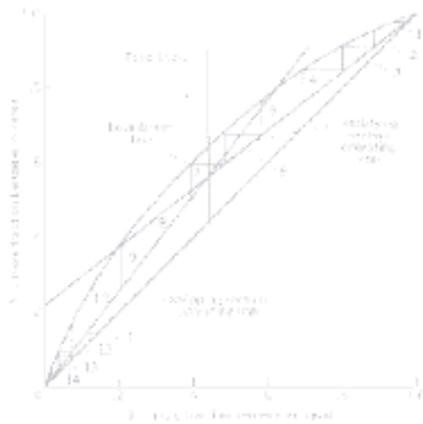
Thus, increasing the number of stages will improve separation.

Effect of Feed Tray Position

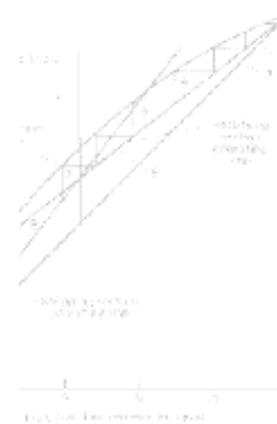
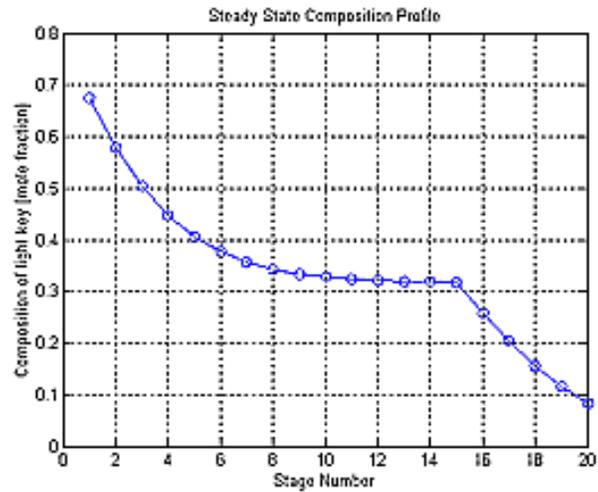
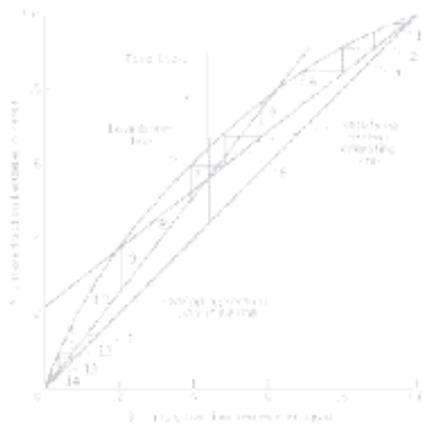
Here we look at how the position of the feed tray affects separation efficiency. Suppose we have a 20 stage column, again separating a binary mixture that has a composition of 0.5 mole fraction in terms of the more volatile component. The terminal compositions obtained when the feed is introduced at stages 5, 10 and 15 (at fixed reflux and reboil rates) are shown in the following plots.



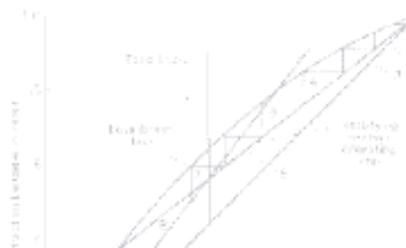
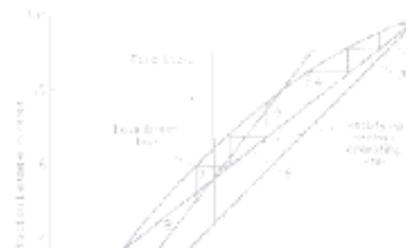
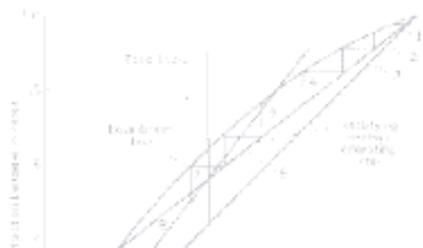
Composition profile: 20 stages, feed at stage 5

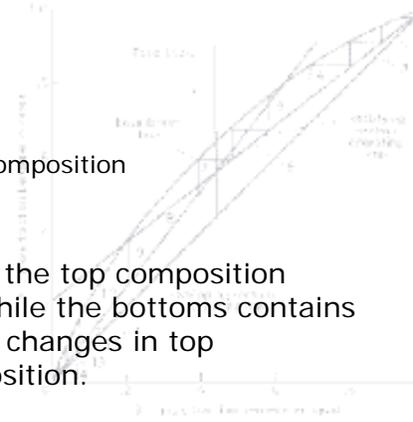
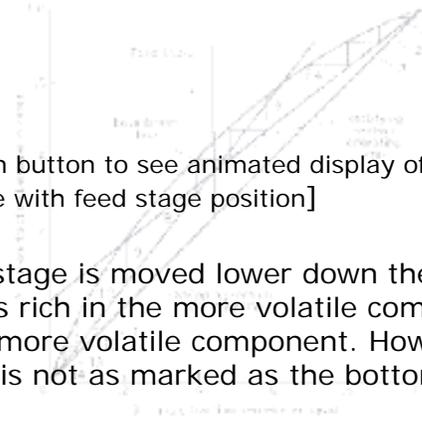
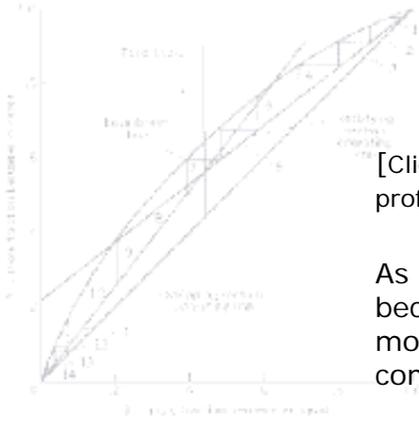
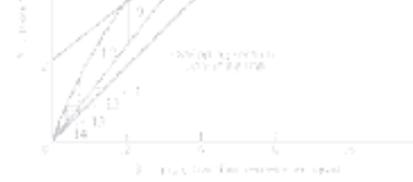
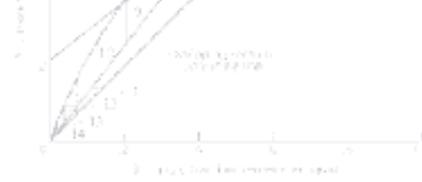
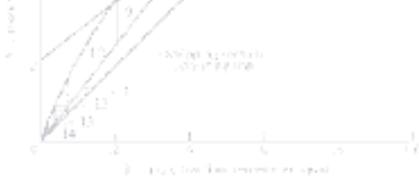


Composition profile: 20 stages, feed at stage 10



Composition profile: 20 stages, feed at stage 15

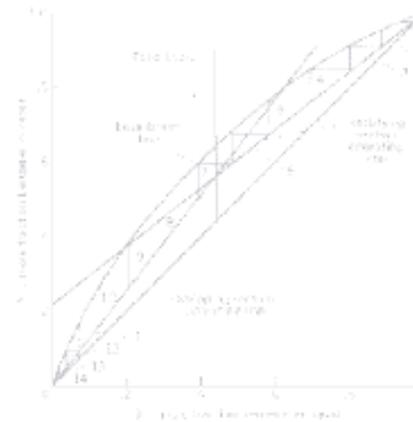
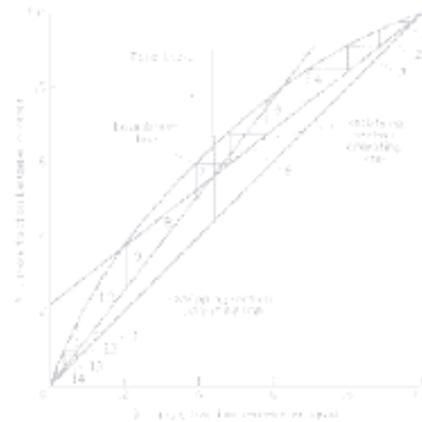
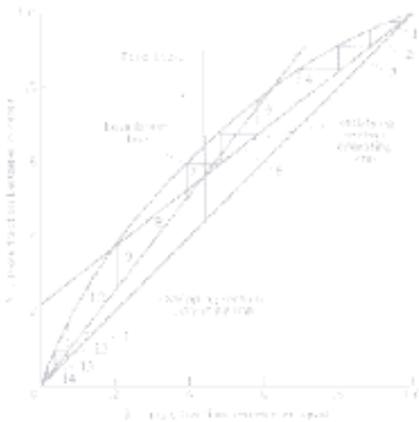
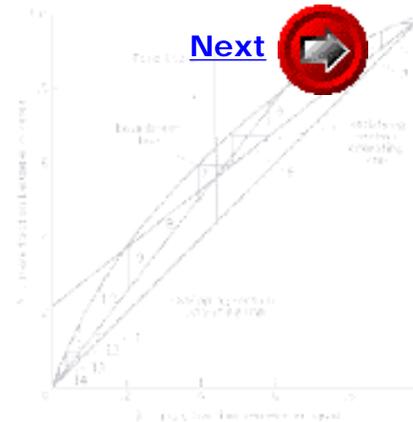
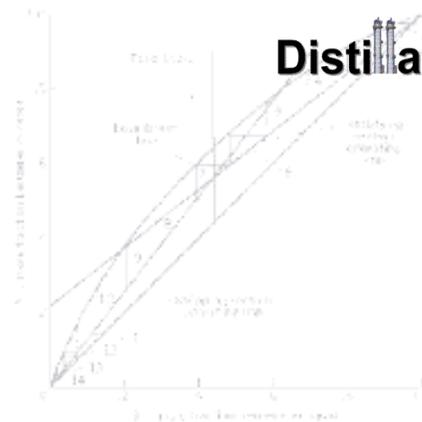
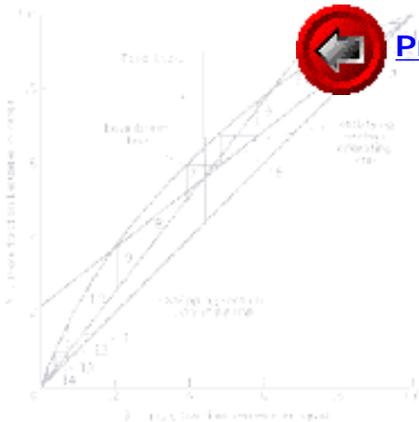




[Click on green button to see animated display of how the composition profiles change with feed stage position]

As the feed stage is moved lower down the column, the top composition becomes less rich in the more volatile component while the bottoms contains more of the more volatile component. However, the changes in top composition is not as marked as the bottoms composition:

The preceding examples illustrate what can happen if the position of the feed tray is shifted for this particular system. They should not be used to generalise to other distillation systems, as the effects are not straightforward.



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FACTORS AFFECTING DISTILLATION COLUMN OPERATION

The performance of a distillation column is determined by many factors, for example:

- ➔ feed conditions
 - state of feed
 - composition of feed
 - trace elements that can severely affect the [VLE](#) of liquid mixtures

➔ internal liquid and fluid flow conditions

➔ state of trays (packings)

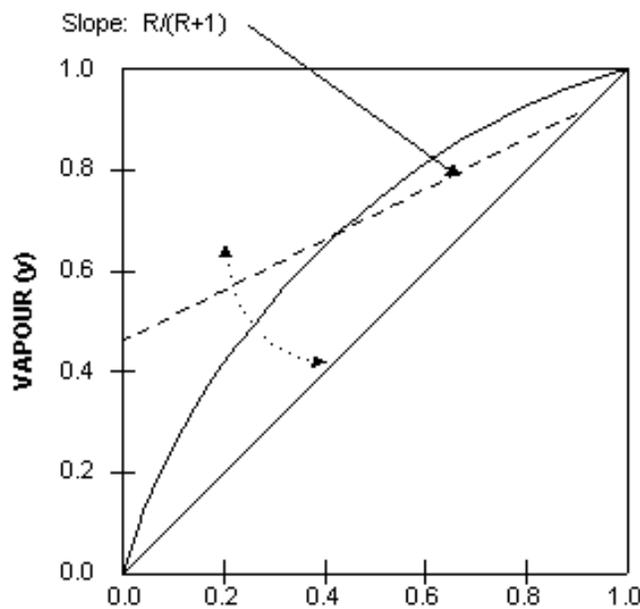
➔ weather conditions

Some of these will be discussed below to give an idea of the complexity of the distillation process.

Feed Conditions

The state of the feed mixture and feed composition affects the operating lines and hence the number of stages required for separation. It also affects the location of feed tray. During operation, if the deviations from design specifications are excessive, then the column may no longer be able handle the separation task. To overcome the problems associated with the feed, some column are designed to have multiple feed points when the feed is expected to containing varying amounts of components.

Reflux Conditions



As the reflux ratio is increased, the gradient of operating line for the rectification section moves towards a maximum value of 1. Physically, what this means is that more and more liquid that is rich in the more volatile components are being recycled back into the column. Separation then becomes better and thus less trays are needed to achieve the same degree of separation. Minimum trays are required under **total**

LIQUID (x)

reflux conditions, i.e. there is no withdrawal of distillate.

On the other hand, as reflux is decreased, the operating line for the rectification section moves towards the equilibrium line. The 'pinch' between operating and equilibrium lines becomes more pronounced and more and more trays are required. This is easy to verify using the [McCabe-Thiele method](#).

The limiting condition occurs at **minimum reflux ratio**, when an infinite number of trays will be required to effect separation. **Most columns are designed to operate between 1.2 to 1.5 times the minimum reflux ratio** because this is approximately the region of minimum operating costs (more reflux means higher reboiler duty).

Vapour Flow Conditions

Adverse vapour flow conditions can cause

- foaming
- entrainment
- weeping/dumping
- flooding

Foaming

Foaming refers to the expansion of liquid due to passage of vapour or gas. Although it provides high interfacial liquid-vapour contact, excessive foaming often leads to liquid buildup on trays. In some cases, foaming may be so bad that the foam mixes with liquid on the tray above. Whether foaming will occur depends primarily on physical properties of the liquid mixtures, but is sometimes due to tray designs and condition. Whatever the cause, separation efficiency is always reduced.

Entrainment

Entrainment refers to the liquid carried by vapour up to the tray above and is again caused by high vapour flow rates. It is detrimental because tray efficiency is reduced: lower volatile material is carried to a plate holding liquid of higher volatility. It could also contaminate high purity distillate. Excessive entrainment can lead to flooding.

Weeping/Dumping

This phenomenon is caused by low vapour flow. The pressure exerted by the vapour is insufficient to hold up the liquid on the tray. Therefore, liquid starts to leak through perforations. Excessive weeping will lead to dumping. That is the liquid on all trays will crash (dump) through to the base of the column (via a domino effect) and the column will have to be re-started. Weeping is indicated by a sharp pressure drop in the column and reduced separation efficiency.

Flooding

Flooding is brought about by excessive vapour flow, causing liquid to be entrained in the vapour up the column. The increased pressure from excessive vapour also backs up the liquid in the downcomer, causing an increase in liquid holdup on the plate above. Depending on the degree of flooding, the maximum capacity of the column may be severely reduced. Flooding is detected by sharp increases in column differential pressure and significant decrease in separation efficiency.

Column Diameter

Most of the above factors that affect column operation is due to vapour flow conditions: either excessive or too low. Vapour flow velocity is dependent on column diameter. Weeping determines the minimum vapour flow required while flooding determines the maximum vapour flow allowed, hence column capacity. Thus, if the column diameter is not sized properly, the column will not perform well. Not only will operational problems occur, the desired separation duties may not be achieved.

State of Trays and Packings

Remember that the actual [number of trays](#) required for a particular separation duty is determined by the efficiency of the plate, and the packings if packings are used. Thus, any factors that cause a decrease in tray efficiency will also change the performance of the column. Tray efficiencies are affected by fouling, wear and tear and corrosion, and the rates at which these occur depends on the properties of the liquids being processed. Thus appropriate materials should be specified for tray construction.

Weather Conditions

Most distillation columns are open to the atmosphere. Although many of the columns are insulated, changing weather conditions can still affect column operation. Thus the reboiler must be appropriately sized to ensure that enough vapour can be generated during cold and windy spells and that it can be turned down sufficiently during hot seasons. The same applies to condensers.

These are some of the more important factors that can cause poor distillation column performance. Other factors include changing operating conditions and throughputs, brought about by changes in upstream conditions and changes in the demand for the products. All these factors, including the associated control system, should be considered at the design stages because once a column is built and installed, nothing much can be done to rectify the situation without incurring significant costs. The control of distillation columns is a field in its own right, but that's another story.



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Distillation

Distillation

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OTHER RESOURCES

General Articles

- [Andrew Sloley's Distillation page](#) contains links to information about the history of distillation processes as well as a glossary of terms.
- [Distillation](#) - a Microsoft Concise Encyclopaedia article
- [Distillation Columns](#) (introductory notes from Cornell Uni.)
- [Energy Conservation in Distillation](#) - from cheresources.com

[Extractive Distillation](#) - a good detailed description of what the method is all about, including the separation of mixtures that form azeotropes.

Reactive Distillation

- [Reactive Distillation](#) - an introduction by T.Mashue and H.S. Fogler (UMich)
- [Website devoted to reactive distillation](#)

Design

- **McCabe-Thiele**
 - [Distillation column design using the McCabe-Thiele method](#) (Stage 1 and above)
 - [On-line McCabe-Thiele calculation of number of theoretical plates](#) (Stage 1 and above)
 - [Calculation of a rectification column](#) - an example from Uni. Koeln, Germany
- [On-line distillation design](#) for separating a single feed stream containing up to 20 components from databank of 480 components.
- [Calculating distillation tray efficiencies](#) has plenty of illustrative photos

Internals

- **Distillation trays**, with some nifty photographs (by tray specialist E. Frank Wijn)
 - [Introduction to trays, in general](#)
 - [Trays, in more detail](#)
- **Packings**
 - [Segmented Packed Columns](#) - see what these guys are capable of
 - [Case studies of the use of packings to improve column performance](#)

Process Control

- [Distillation column control design using steady state models](#) - describes

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the use of steady models to come up with a practicable distillation column control strategy (by Hyprotech)

- [Pressure control of distillation columns](#) by Andrew Sloley

Other Links

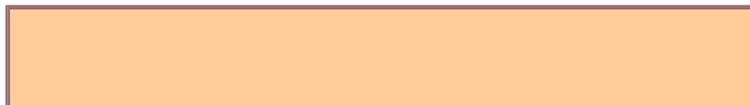
- [Distillation resources](#) from miningco.com



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