Basic Operator Training Course

Distillation Fundamentals

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Basic Operator Training Course

Distillation Fundamentals

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Distillation Fundamentals

Module Objectives

1. Explain the differences between boiling and condensing for pure substances and mixtures
2. Describe single stage distillation as a tool to illustrate the principles of distillation
3. Describe the relationship between temperature and composition in distillation
4. Apply the principle of a single stage distillation to achieve higher purity separation
5. Explain the purpose and function of reflux and reflux ratio
6. Distinguish between the different modes of operation for distillation
7. Summarize equilibrium in distillation
8. Demonstrate how to find composition in a phase diagram
9. Identify elements and flow for a distillation system
10. Explain the design and function of trays and other column internals
11. Describe equilibrium in relation to column trays and associated temperature, pressure, and composition relationships and variables that affect distillation
12. Describe heat and material balance for distillation systems
13. Explain fractionation and vacuum distillation
14. List the methods used to evaluate performance of a distillation

Module Introduction

Distillation is a thermal separation process based on vaporization and condensation. It is widely used in refineries and some chemical plants. A mixture of two or more components can be separated when the boiling points of the component in a mixture are significantly different. Distillation is typically preformed in distillation columns.

Module Overview

This module first introduces the physical principles of distillation. The significance of boiling points, temperature, pressure, and composition is explained. From there, a closer look at distillation systems and types of distillation columns describes how distillation works on an industrial scale. Important operating parameters for distillation, such as material and heat balances, are analyzed.

Term	Description
Batch Mode	An operating mode where he process has a start and an end. Batch distillation means, all feed material is exposed to heat at the same time; once the desired amount is vaporized/condensed, heat supply is removed.
Boiling Point	The temperature at which a pure substance vaporizes. The temperature remains constant until all of the substance is vaporized.
Bubble Point	The temperature at which the first vapor bubble forms in a mixture.
Composition	The quantification of the amount of components in a mixture.
Condensation	The process of turning vapor into liquid.
Continuous Mode	An operating mode where feed and products flow continuously.
Cut Point	The temperature at which two components separate by distillation.
Dew Point	The temperature at which the first vapor bubble condenses from a vaporized mixture.

Term	Description
Distillation	A separation process based on the difference in boiling points of separated components.
Distillation System	All equipment associated with a specific distillation process.
Downcomer	A column internal attached to a tray; a channel that allows liquid to flow from one tray to the tray below.
End Boiling Point (EBP)	A quality measurement of distillation products. The temperature at which a product sample is completely vaporized. It indicates how much of the ***heavier*** component is in a product.
Equilibrium, Vapor-Liquid Equilibrium	Equilibrium in distillation means the amount of vaporization is the same as the amount of condensation. The temperature and composition in the liquid and in the vapor do not change over time.
Ejector	A piece of equipment used to draw a stream from equipment by creating a suction pressure.
Fraction	A portion of a mixture; a fraction has properties that allows for separation from other fractions.

Term	Description
Heavier Component	Indicates that this component has a higher boiling point and higher molecular weight than other components in a mixture; typically used for hydrocarbons.
Knockout Tray	Column internal; collects falling liquid and to route it to specific destinations; used for side product draws.
Initial Boiling Point (IBP)	A quality measurement of distillation products. The temperature at which a product sample starts boiling. It indicates how much of the ***lighter*** component is in a product.
Latent Heat	Heat supply that generates a phase change, such as when water vaporizes or condenses.
Lighter Component	Indicates that this component has a lower boiling point and lower molecular weight than other components in a mixture; typically used for hydrocarbons.
Mixture	A substance containing at least two types of different molecules.

Term	Description
Mole Fraction	The number of moles of one component in proportion to the total number of moles in the mixture.
Multi-Stage Distillation	Consecutive repetition of single stage distillation. A distillation column itself can be viewed as multi-stage distillation with each tray being a stage.
Overhead Condenser	Part of a distillation system; condenses the vapor that exits the top of the column.
Overhead Receiver	A vessel that collects condensed overhead vapor from a distillation column and allows for separation of uncondensed vapor.
Packing	A column internal used to intensify vapor-liquid contact.
Phase Change	A change of state of matter by applying or removing heat.
Pure Substance	In the context of this module, a pure substance is a gas or liquid that contains only one type of molecule (or atom).

Term	Description
Pumparound	A side draw stream from a distillation column that is cooled and returns to the column to provide reflux.
Reboiler	The main heat source for a distillation system; receives bottoms liquid, vaporizes it, and returns vapor to the column. The reboiler generates most vapor that rises through a distillation column.
Reboiler Duty	The amount of heat supplied by the reboiler to the distillation column.
Reflux	A cold stream of condensed overhead vapor that returns to the distillation column. Reflux provides cooling to a distillation column and is important for temperature control of the column.
Reflux Ratio	The ratio of reflux flow and overhead product(s) flow. The reflux ratio is used to control the purity of overhead product.
Reid Vapor Pressure (RVP)	A quality measurement of distillation products. It indicates the volatility of gasoline and other crude oil fractions.

Term	Description
Semi-Batch Mode	An operating mode where either feed or product(s) flow continuously.
Sensible Heat	Heat supply that creates a change in temperature, such as when the water temperature is raised to bring water to boiling.
Single Stage Distillation	A model used to describe the process of vaporization/ condensation and mass transfer on a single distillation tray.
Still	A distillation apparatus that is used for distillation, consisting of equipment for vaporizing a mixture and condensing/collecting the vaporized material. There is no reflux flow.
Thermosiphon	Pumpless circulation of liquid based on density/gravity and phase change. A thermosiphon reboiler receives liquid from the column bottom; the heat supply vaporizes liquid, which causes more liquid to flow from the column into the reboiler to replace the volume lost to vapor.
Vacuum	Pressure below atmospheric pressure.

Term	Description
Vaporization	The process of turning liquid into vapor.
Vapor Pressure	Pressure exerted by vapor bubbles in a mixture of liquid and vapor.
Volatility	The tendency of a substance to vaporize.

Distillation Fundamentals

Distillation Principles

Chapter Overview

Vaporization and condensation are the processes that drive distillation. This chapter takes a closer look at the dynamics of vaporization and condensation of pure substances and mixtures. These concepts are utilized in a single stage distillation, a tool to simplify and explain what happens in distillation.

At its core, distillation takes advantage of the differences in relative volatilities of a mixture's components. Applying heat to a two-component mixture will vaporize the more volatile component, while leaving the less volatile component in the liquid phase.

Chapter Objectives

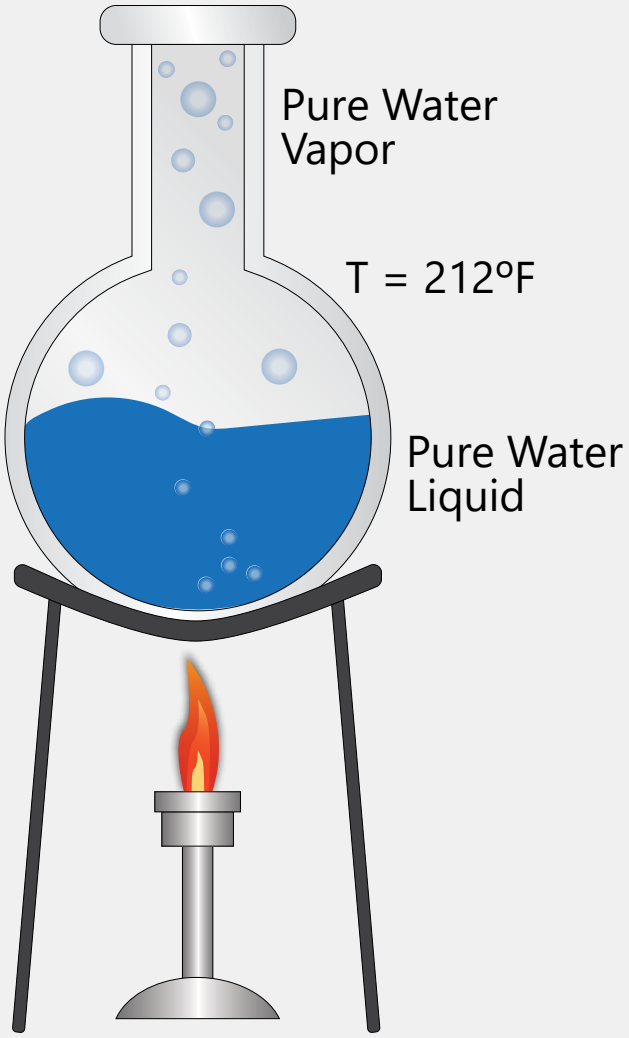
1. Explain the differences between boiling and condensing for pure substances and mixtures
2. Describe single stage distillation as a tool to illustrate the principles of distillation
3. Describe the relationship between temperature and composition in distillation
4. Apply the principle of a single stage distillation to achieve higher purity
5. Explain the purpose and function of reflux and reflux ratio
6. Distinguish between different modes of operation for distillation
7. Summarize equilibrium in distillation

Boiling and Condensing Pure Substances

A pure substance in the context of this course is matter (liquid or vapor) that contains only one type of molecule or one component. The knowledge of how pure substances boil, vaporize, and condense is necessary to later understand how mixtures vaporize and condense.

Boiling Point of a Pure Substance

The **boiling point** of a pure substance is the temperature at which a liquid starts to ***vaporize***. During vaporization, the temperature of the boiling pure substance does not change. For example, water starts to boil at 212°F (at atmospheric pressure), but the temperature of the boiling water remains at 212°F during vaporization despite the continuous heat supply. ***The applied heat generates a phase change, not a temperature change.***



Vaporizing Water

Boiling and Pressure

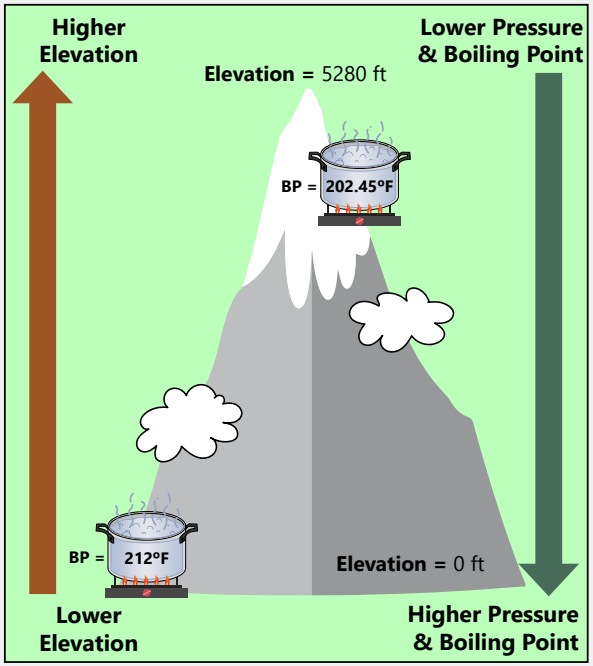
The boiling point strongly depends on **pressure**. If the substance is exposed to a higher pressure, boiling will start at a higher temperature.

Conversely, lowering the pressure will reduce the boiling point temperature.

This can be explained by the concept of **vapor pressure**: the boiling point is reached when the pressure of vapor bubbles is high enough that vapor can escape the liquid. In other words, the pressure of the vapor bubbles is equal to the surrounding pressure.

At high elevations, water boils at a much lower temperature than 212°F because the atmospheric pressure is lower.

This same principle is utilized by pressure cookers; at higher pressure, boiling occurs at a higher temperature, which in consequence speeds up cooking.



Boiling Point (BP) and Pressure

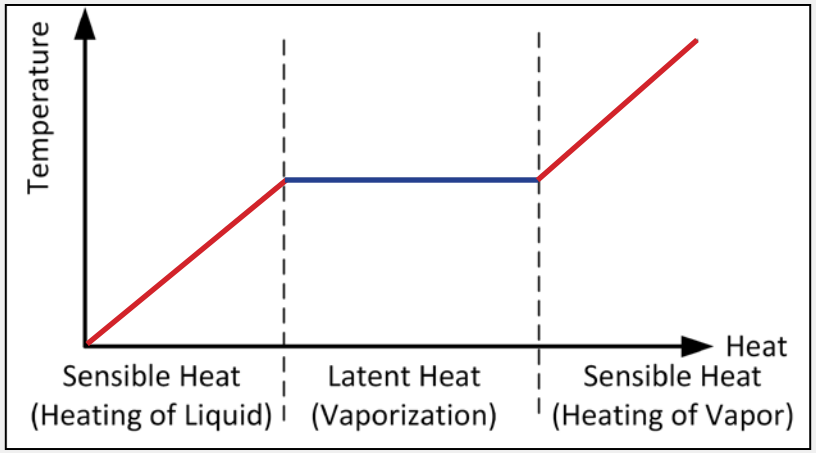
Heat and Phase Changes

Heat supply that creates a change in temperature, such as when the water temperature is raised to bring water to boiling, is called **sensible heat**.

When all liquid is vaporized and more heat is supplied (and only then), the vapor temperature increases; this is also sensible heat.

Heat supply that generates a phase change, such as when water vaporizes or condenses, is called **latent heat.** The temperature remains constant.

The graphic shows how sensible heat supply causes changes in temperature, while latent heat supply does not change temperature.



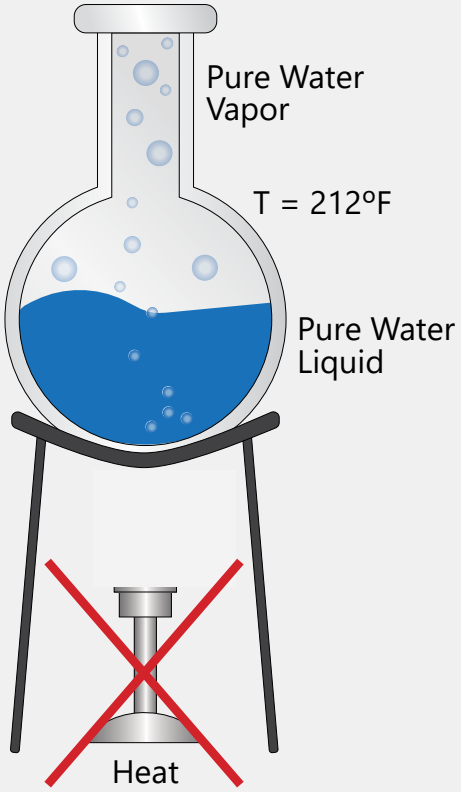
Sensible and Latent Heat

Condensation of a Pure Substance

When no more heat is applied to a boiling substance, **condensation** occurs.

The temperature at which condensation of a pure substance occurs is the same as the substance's boiling point temperature. Until all vapor is condensed, the temperature does not change.

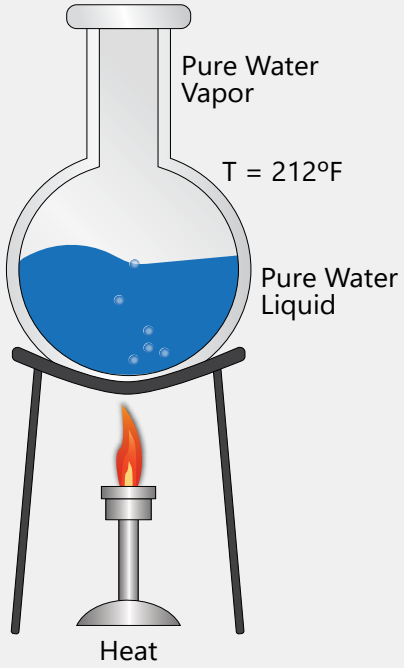
For example, if water has been partially vaporized at atmospheric pressure and the heat source is removed, the vapor condenses. Until all vapor is liquefied, the temperature will remain at 212°F.



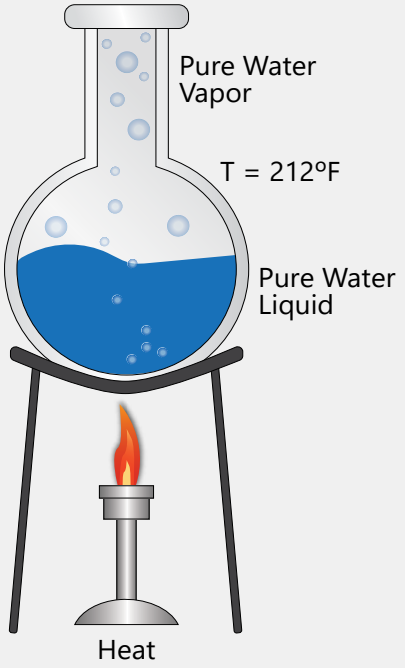
Vapor Pressure

The graphics below depict how vapor pressure and boiling are related.

In the left graphic, vapor bubbles form but the atmospheric pressure is higher than the bubble pressure; bubbles can not escape the liquid. In the right graphic, boiling has progressed; the bubbles' pressure has overcome the atmospheric pressure, and bubbles can escape.



Low Vapor Pressure



High Vapor Pressure

Boiling and Condensing Mixtures

A mixture in the context of this course is matter (liquid or vapor) that contains at least two types of molecules or components. The knowledge of how mixtures boil, vaporize, and condense is necessary to later understand how mixtures can be separated into components.

Boiling Point of a Mixture

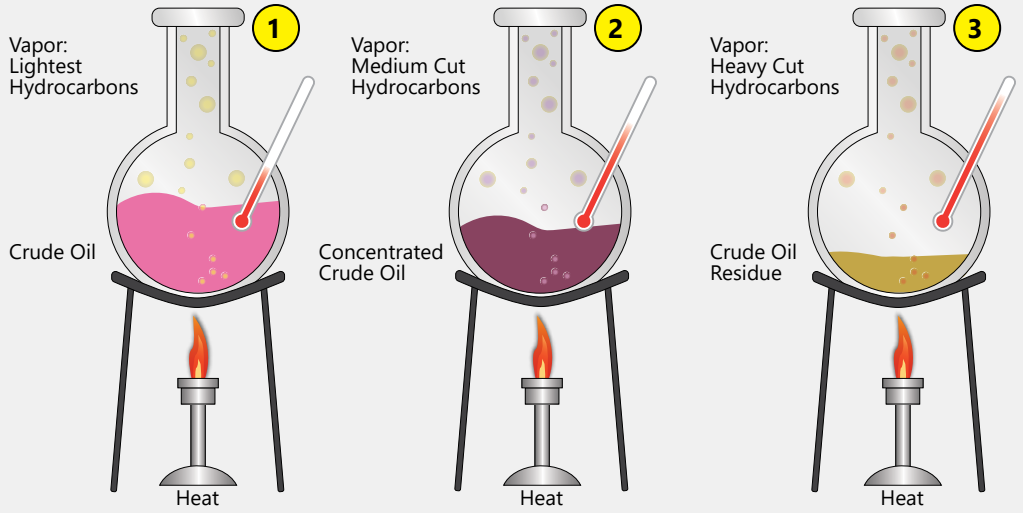
Unlike pure substances, **mixtures** have ***multiple boiling points***. When heat is applied to a two component (binary) mixture, the mixture starts boiling at a temperature specific to the composition of this mixture. ***More of the more volatile component in a mixture means a lower boiling point, and vice versa.***

The boiling point of the mixture is somewhere between the boiling points of the pure individual substances. The component with the lower boiling point vaporizes first; in other words, it is more ***volatile***. As the more volatile component vaporizes, the concentration of the less volatile component in the mixture increases. As a result, the boiling point of the mixture will increase. As the more volatile component continues to boil off, an increase in temperature can be observed. When all of the more volatile component is vaporized, the temperature will reach the boiling point of the less volatile component and will not change anymore. The temperature at which the first vapor bubble forms in this mixture is called the **bubble point**.

Example: Boiling Crude Oil

The graphics to the right illustrate the change in temperature due to boiling points for crude oil components; crude oil is a mixture of multiple hydrocarbons with different boiling points.

1. When a high enough temperature is reached, the lightest, most volatile component vaporizes. The temperature continuously increases because increasingly less of this light material is in the liquid.
2. All of the lightest material is vaporized. The temperature is now at a value that allows the medium cut hydrocarbons to vaporize; these have a higher boiling point than the previously vaporized components. The temperature continues to increase as the medium cut hydrocarbons boil off.
3. The temperature is at a value where all of the medium cut material is vaporized. The heavy cut material vaporizes.



Condensing a Mixture

Unlike pure substances, mixtures condense at multiple temperatures.

For this example, assume that a mixture of two components is at a temperature where the more volatile component is completely vaporized as well as some of the less volatile component.

When heat is removed, the temperature falls. The less volatile component condenses first because it has a higher boiling point. It becomes liquid when the vapor is cooled to its boiling point and below.

With further cooling, the temperature will fall to the point where the more volatile component condenses; it has the lower boiling point and liquefies when cooled to its boiling point and below.
The temperature at which the first vapor bubble condenses from a vaporized mixture is called the **dew point.**

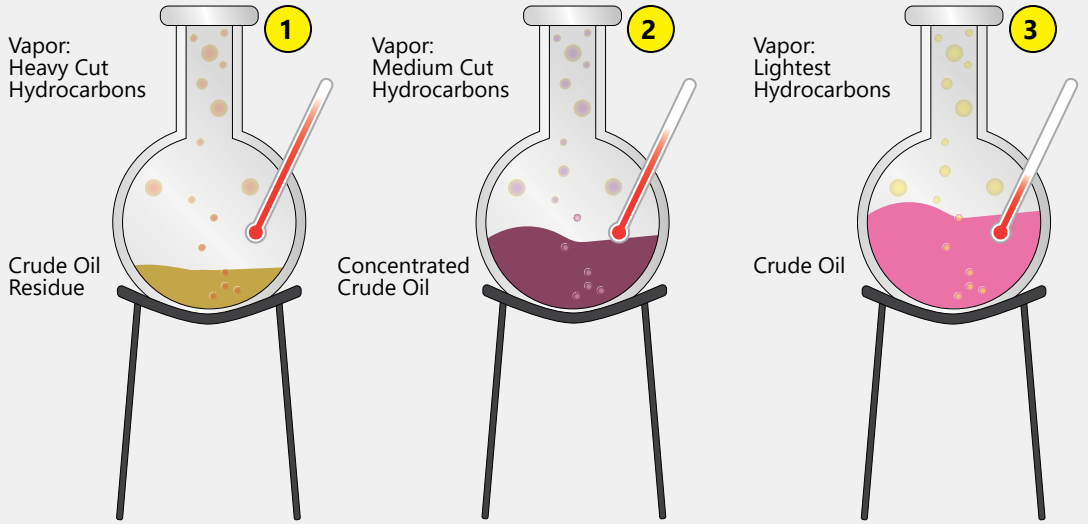
Example: Condensing Crude Oil

The graphics to the right illustrate how components of crude oil condense at different temperature when the heat source is removed.

1. When the heat source is removed from the previously vaporized mixture, the temperature falls to the boiling point of the heavy cut hydrocarbons. This material condenses.

2. When all of the heavy cut hydrocarbons are condensed and the temperatures falls further, the medium cut hydrocarbons condense.

3. When all of the medium cut hydrocarbons are condensed and the temperature falls further, the lightest hydrocarbons condense.



Vaporizing to Separate a Mixture

The fact that the more volatile component starts vaporizing first when heat is supplied to a mixture allows you to separate the more volatile component from the mixture.

Vaporizing to Separate a Mixture

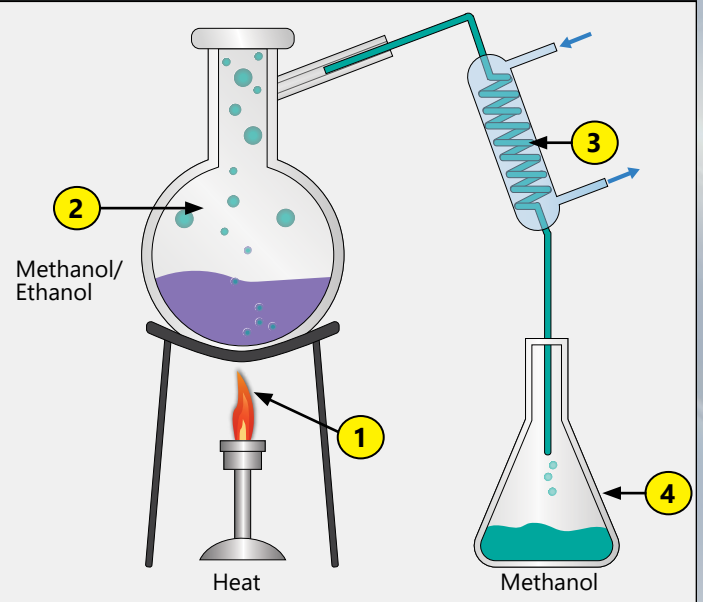
The graphic shows the principle of separating a mixture by vaporizing. This arrangement is sometimes called a **still**.

1. Heat is supplied to the methanol/ethanol mixture contained in a flask. *Methanol has a lower boiling point than ethanol.*

2. When the temperature reaches the boiling point of the mixture, vaporization begins. Vapor separates from the liquid and rises upward. Methanol vaporizes first because, as previously mentioned, it has a lower boiling point than ethanol. The temperature in the liquid continuously increases due to the increasing concentration of ethanol.

3. Methanol vapor exits and flows through a coil that is cooled using cooling water.

4. As a result, the methanol vapor condenses and collects in a separate container.



Boiling Point Methanol: 148.5˚F

Boiling Point Ethanol: 172.6˚F

If heat supply continues, eventually all of the methanol can be vaporized. *The temperature in the flask continuously increases, and it is always equal to the boiling point of the composition of the liquid mixture.* The supplied heat creates a phase change. This heat is called **latent heat**.

When all of the methanol is vaporized from the flask, the supplied heat causes the temperature to increase to the boiling point of pure ethanol. The heat needed to raise the temperature until this vaporization occurs is called **sensible heat**. If heat supply is not stopped, ethanol will boil off and also enter the receiving flask.

If heat supply is stopped at just at the point when all methanol is vaporized, this will result in essentially complete separation (nearly pure components). In reality however, some methanol will remain with ethanol and some ethanol will boil off with methanol. The impurity of the two separated materials can be unsatisfying. To combat this, a step must be added, which will be explained on the following page.

Single Stage Distillation

Single stage distillation is a concept describing one stage of distillation. The difference to the previously discussed process is that reflux stream is added to the system.

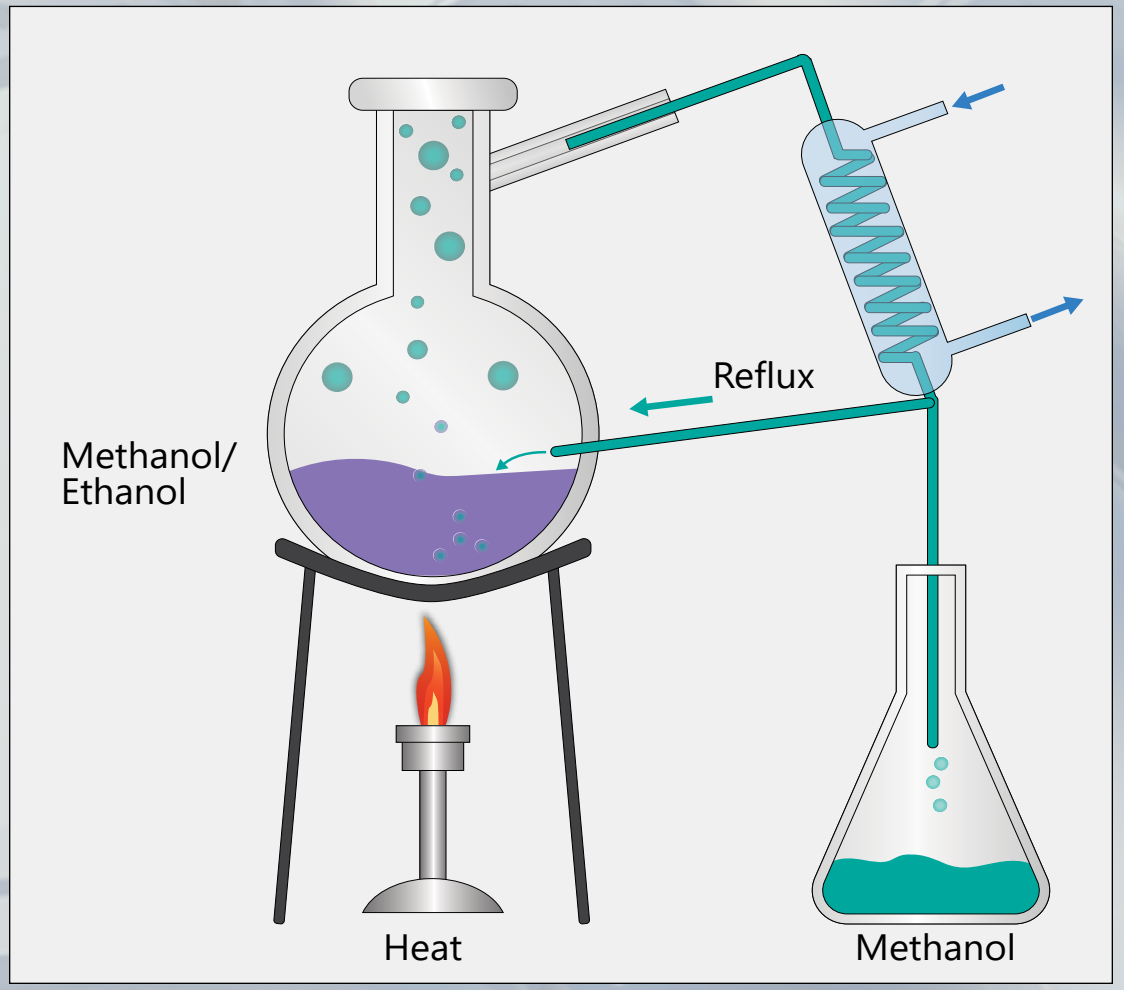
Concept of Single Stage Distillation

Single stage distillation adds a condensation step to the previously shown vaporization process (still). Condensation is accomplished by using a reflux stream.

Reflux is condensed boiled off material. It is returned to the vapor phase in the boiling flask.

Returning reflux to the heated flask results in the following:

- The cooled liquid reflux condenses a portion of the hot vapor
- The component with the higher boiling point (ethanol) condenses and falls back into the boiling liquid
- The methanol contained in the liquid reflux vaporizes again
- The result is ***purer methanol and purer ethanol***



Function of Reflux

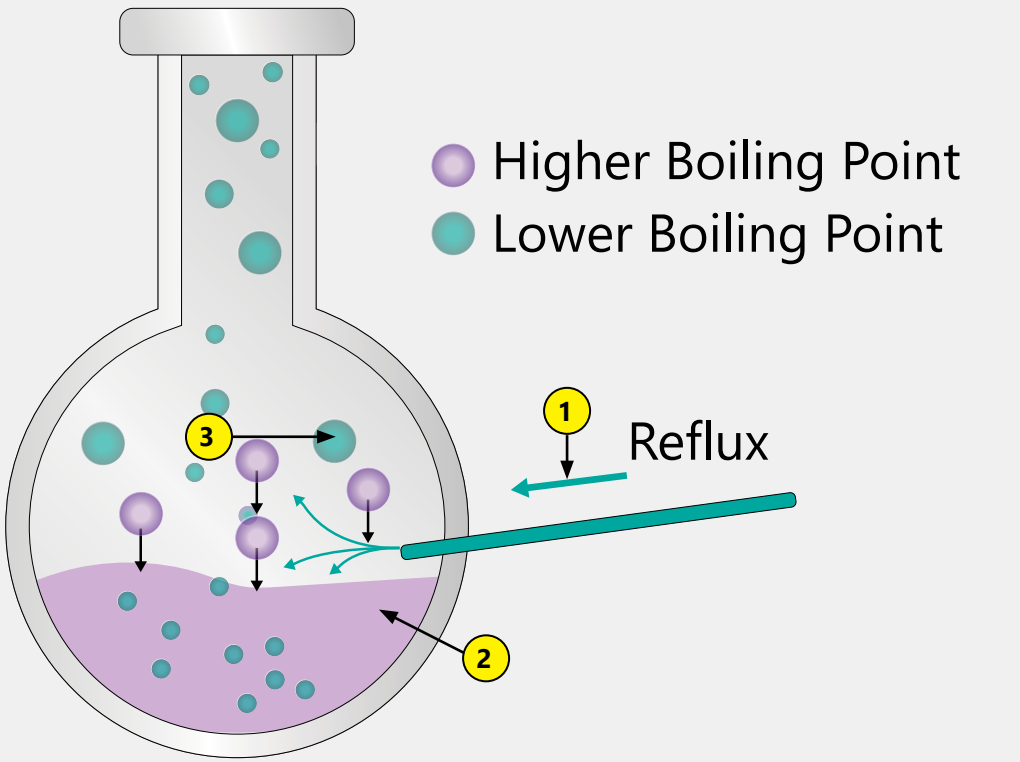
Let's take a closer look at the role of reflux.

The reflux contains ***mostly the lighter material, but also some of the heavier material.***

1. When the reflux returns into the boiling flask, heat exchange occurs between the hot vapor that rises in the flask and the cooler reflux. This heat exchange causes mass transfer.

2. The cool reflux condenses some of the heavier material contained in the vapor. The condensed vapor falls back into the heated flask.

3. The hot vapor in the flask vaporizes the lighter portion of the reflux. This material exits together with the vapor created by the heat supply to the flask.



Temperature and Composition

Temperature is one of the main variables in distillation and has a large effect on the quality of separated products. ***Temperature is strongly correlated to composition***.

Any change in composition results in a temperature change. Any change in temperature results in a change in composition.

Example: Methanol/Ethanol

Consider the following mixtures of methanol (M) and ethanol (E), as shown on the right

A mixture containing 90% M and 10% E has a ***lower*** boiling point than a mixture containing 10% M and 90% E. This is because methanol is more volatile (lower boiling point) than ethanol.

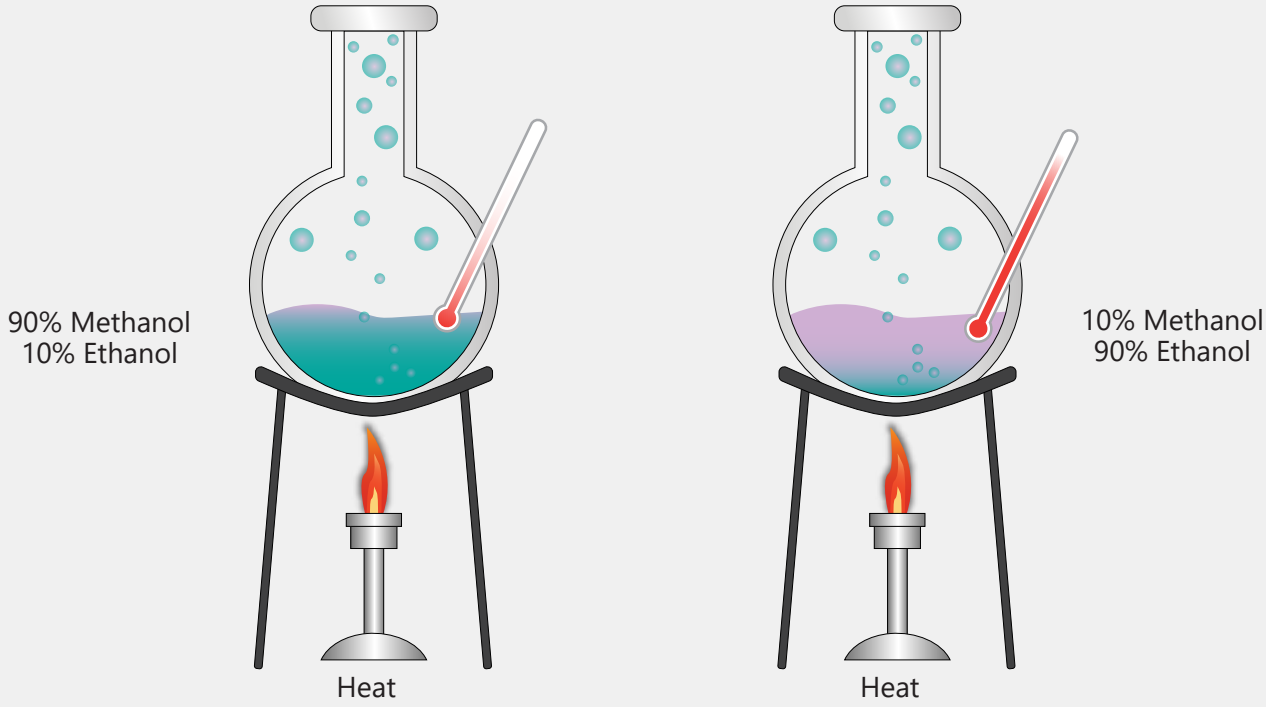
Any change in temperature indicates a change in composition of the vapor and liquid phases.
Over the course of the distillation process, the methanol concentration in the boiling liquid decreases and the ethanol concentration increases.

This can be observed by a change in temperature in the boiling flask: ***the higher the temperature, the higher the concentration of ethanol in the boiling liquid.***

The graphics on the following page illustrate how temperature and composition are related.

Boiling Point Methanol: 148.5˚F

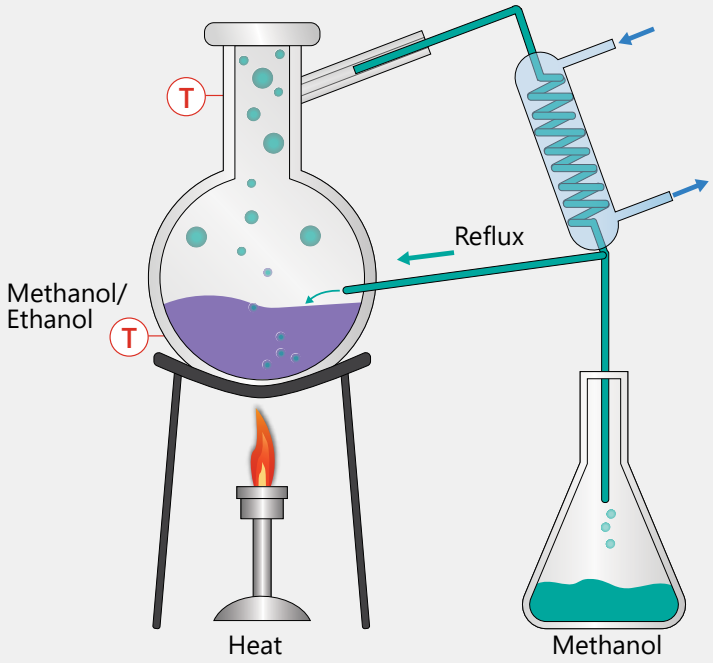
Boiling Point Ethanol: 172.6˚F



Composition and Temperature over Time

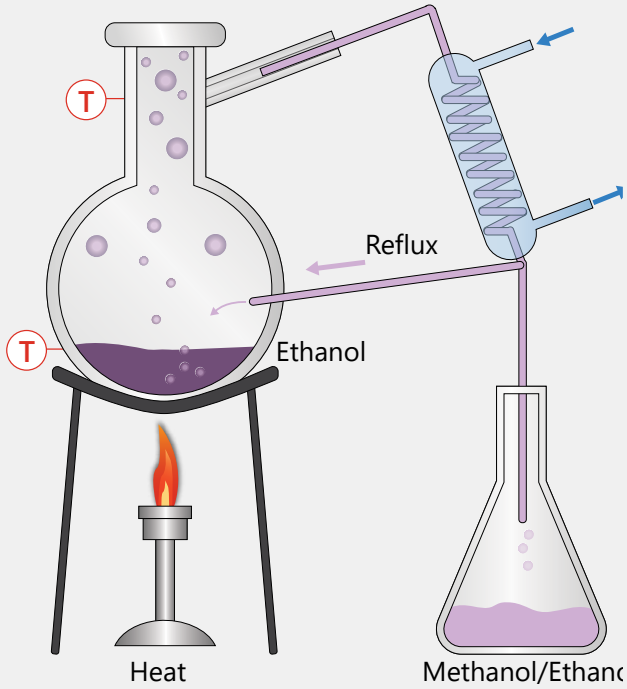
Time 1

Temperature = Boiling Point of Mixture



Time 2

Temperature = Boiling Point of Ethanol



The left graphic shows a specific moment (Time 1) during this batch distillation. Heat is applied, the mixture starts boiling, and the lighter component (methanol) vaporizes.

Inside the flask, the temperature of the boiling liquid is the same as the temperature of the vapor. This temperature is the ***boiling point of the current liquid mixture.***

The composition in the vapor phase is different than that of the liquid phase. Vapor contains mainly methanol; liquid is a mixture of methanol/ethanol.

The right graphic shows the same process at a later time (Time 2): all of the methanol has boiled off. The composition of the boiling liquid is 100% ethanol.

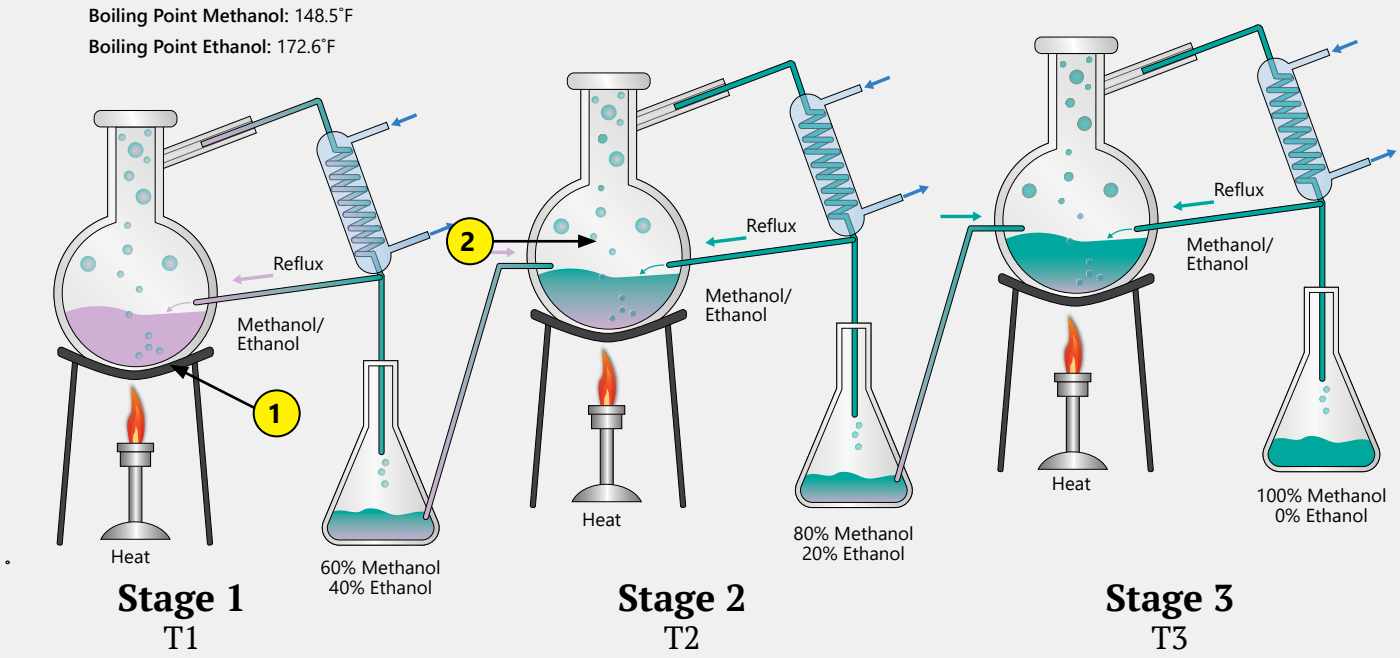
As stated in the previous example (Time 1), the temperature of the boiling liquid is the same as the temperature of the vapor. Now, at Time 2, this temperature is ***the boiling point of ethanol***.

The composition in the vapor phase is now the same as in the liquid phase. This is the point of simple ethanol vaporization; no more distillation occurs.

Multi-Stage Distillation

If the process of single stage distillation is consecutively repeated, it is called multi-stage distillation. Repeating the process of single stage distillation results in a purer product.

Concept of Multi-Stage Distillation



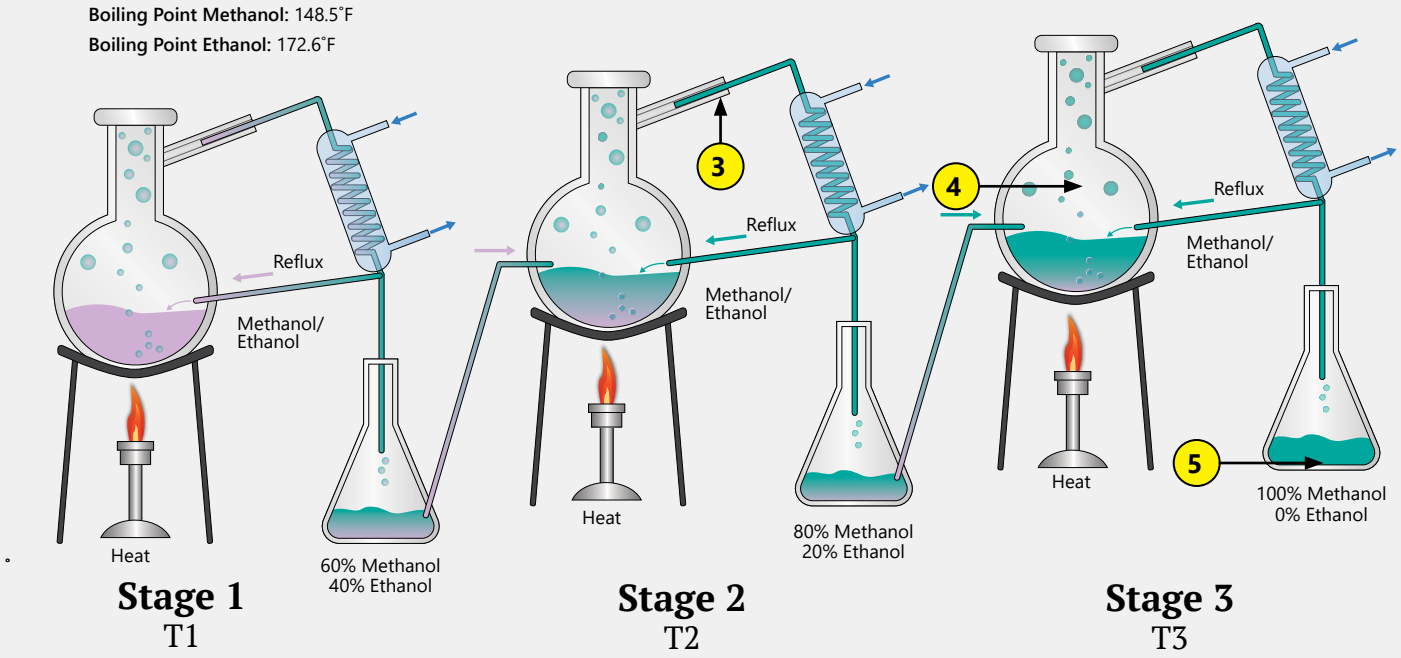
Boiling Temperatures: T1 > T2 > T3

The graphic depicts how the principle of a single stage distillation can be utilized in industrial distillation. The single stage distillation is repeated ***multiple times***.

1. The first single stage distillation boils at temperature T1. The condensed vapor in the collection flask still contains a large amount of ethanol in methanol.

2. The condensed liquid from the first stage is sent into a second stage. Because the concentration of ethanol is lower in this liquid, it boils at a lower temperature (T2).

Concept of Multi-Stage Distillation (cont.)



Boiling Temperatures: T1 > T2 > T3

3 The second distillation stage separates vapor with higher methanol purity, but the condensed vapor in the collection flask still contains some undesired amounts of ethanol.

4 The condensed liquid from the second stage again undergoes a third distillation stage. The boiling temperature (T3) is lower than the previous flasks due to the lower ethanol content.

5 In the third stage, pure methanol is boiled off and condensed. The final product is pure methanol.

In this example, the boiling temperature in the heated flasks ***decreases from left to right*** because the boiling liquid contains ***increasingly more of the more volatile component.***

That being said, achieving 100% pure separation is only theoretical. Even with the most advanced distillation, each separated product will always contain some of the other component. ***The more distillation stages that are used, the more pure the substances become***. This principle is applied to distillation columns by adding trays or packing.

Modes of Operation

Distillation in the industry can be performed using one of three operating modes. The mode of operation is selected based on operational requirements such as how much product must be distilled.

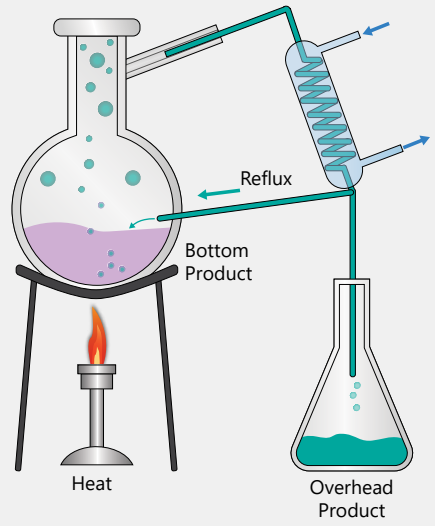
Batch Mode

Batch mode, shown below, means that ***all of the mixture is placed in a vessel at once***.

Heat is applied to vaporize the more volatile component. When the temperature starts rising towards the boiling point of the less volatile component, heat supply is removed and the mixture is considered separated.

The more volatile component is condensed in the collection flask, and the less volatile component remains in the original vessel.

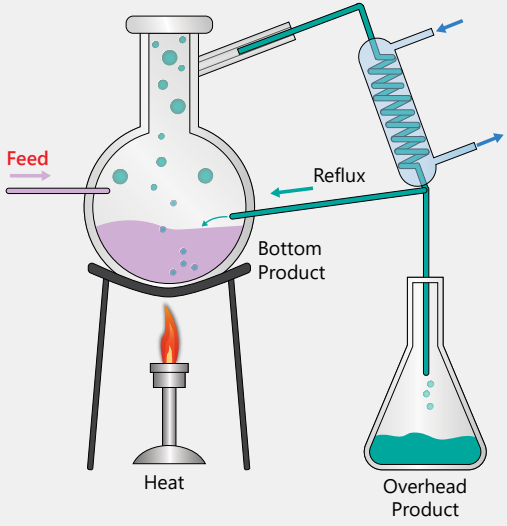
Batch or still distillation is used for ***smaller or infrequent production***.



Semi-Batch Mode

Semi-batch mode, shown below, means that the mixture is placed in a vessel like in batch mode. As a portion of the mixture vaporizes, ***additional feed*** is supplied to the vessel.

Semi-batch mode is used when ***the amount of mixture is too large to be placed in the vessel at once.*** It is also used when not all of the mixture can be heated to boiling point at once.

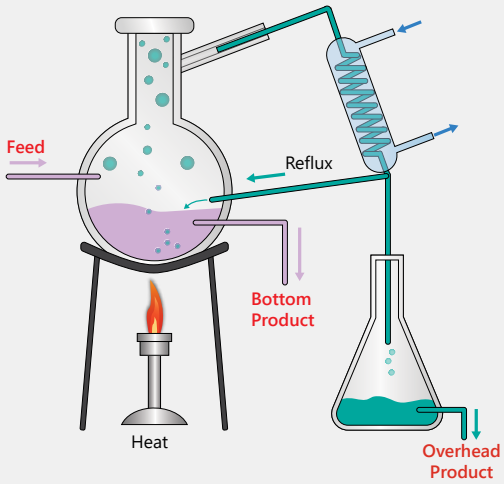


Continuous Mode

Continuous mode, shown below, means that ***feed is continuously supplied*** to the heated vessel. In addition, ***both products are continuously removed***.

To maintain the amount of mixture in the heated vessel, the products are removed in proportion to the feed rate and composition.

Continuous mode is the most common industrial application.



Vapor-Liquid Equilibrium

In a continuous distillation process, the liquid and the vapor above the liquid are in an equilibrium. This means that the composition of vapor and liquid do not change over time in steady operation.

Vapor-Liquid Equilibrium

In distillation, the vapor phase and the liquid phase have ***different compositions***. As shown in the previous examples, there is more methanol in the vapor phase than in the liquid phase. More ethanol is in the liquid phase than in the vapor phase.

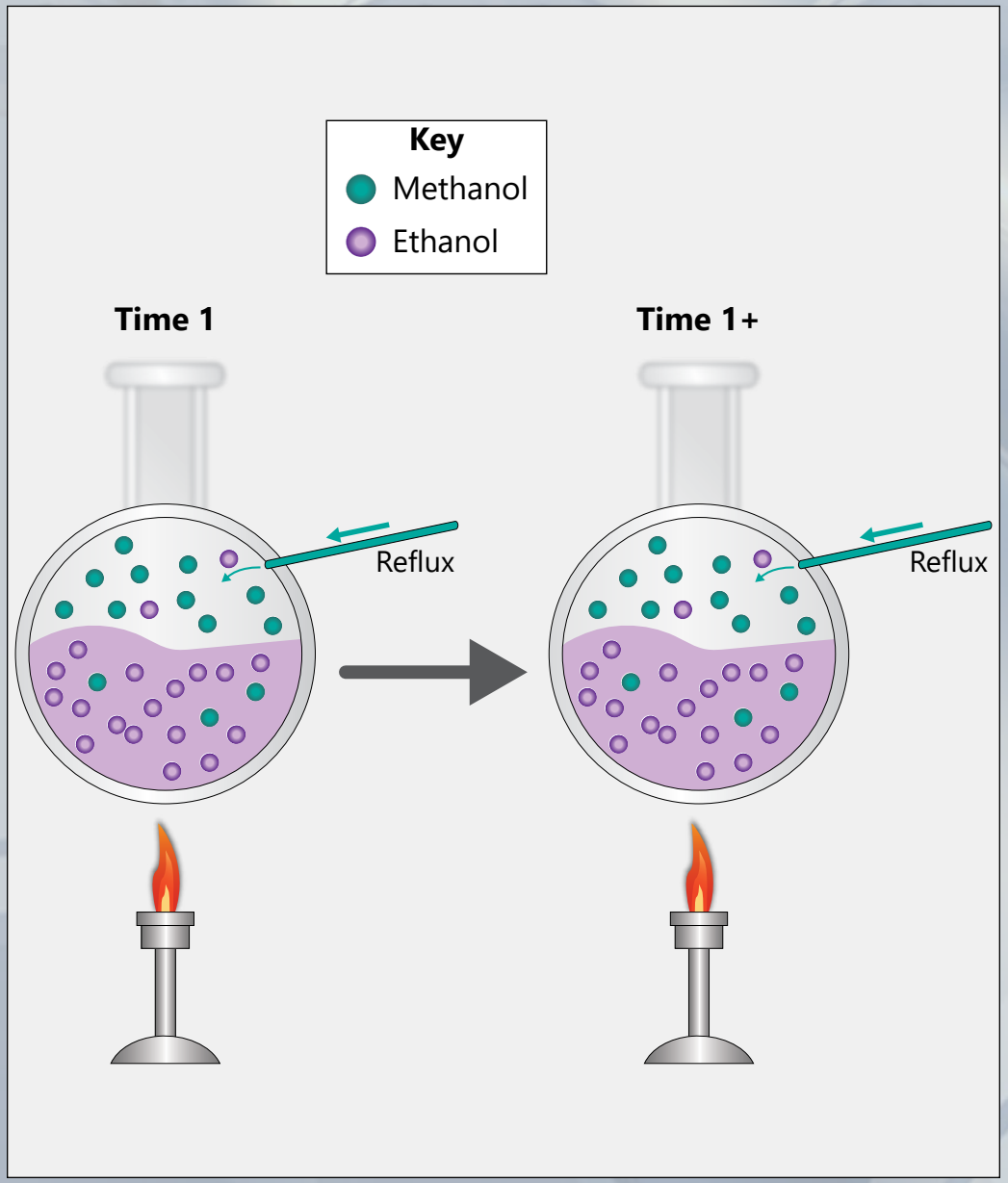
In a continuous distillation with reflux, the vapor and liquid phase reach a **vapor-liquid equilibrium**. ***Equilibrium in distillation means the amount of vaporization is the same as the amount of condensation.*** The temperature and composition in the liquid and in the vapor do not change. That does ***not*** mean that the composition in the liquid and vapor are equal.

The example to the right depicts what this equilibrium means:

Consider the same flask of boiling methanol/ethanol as part of a continuous distillation process as before. At a specific time (Time 1), the liquid contains many more ethanol moles than methanol moles. The vapor contains many more methanol moles than ethanol molecules.

After some time has elapsed (Time 1+) and the temperature remains unchanged, the composition of the liquid phase is the ***same*** as before. The composition of the vapor phase is also the ***same***.

However, despite the equilibrium, the composition of the vapor phase is ***different*** than the composition of the liquid phase.



Notes

Discuss with a Trainer

The temperature at which a pure substance vaporizes is called the _____.

The bubble point of a mixture is the temperature at which _____ _____.

Heat that causes a phase change of a substance is called _____ _____.

Batch distillation means _____ _____.

The boiling point of substance increases when the pressure ____ _____.

Continuous distillation means _____ _____.

Discuss with a Trainer

Vapor-liquid equilibrium in distillation means that the amount of condensation is _____ the amount of vaporization.

The source of reflux is material that _____.

Distillation Fundamentals
Industrial Distillation Systems

Chapter Overview

Industrial applications of distillation require a distillation system. The heart of the system is a distillation column. Within the distillation column, the process of single stage distillation is utilized and multiplied. Distillation columns also contain internals that are specifically designed to promote the separation of a mixture.

A distillation system also requires heating and cooling equipment to aid in the separation process. This chapter describes the process in a distillation system, how the physical principles discussed in the previous section are applied, and how different variables affect distillation.

Chapter Objectives

1. Identify elements and flow for a distillation system
2. Explain the design and function of trays and other column internals
3. Describe equilibrium in relation to column trays and associated temperature, pressure, and composition relationships and variables that affect distillation
4. Describe heat and material balance for distillation systems
5. Explain fractionation and vacuum distillation
6. List methods used to evaluate performance of a distillation

Typical Distillation System

Most distillations are performed in continuous mode. These processes are all equipped with the same basic equipment and operate in the same manner.

Equipment and Function

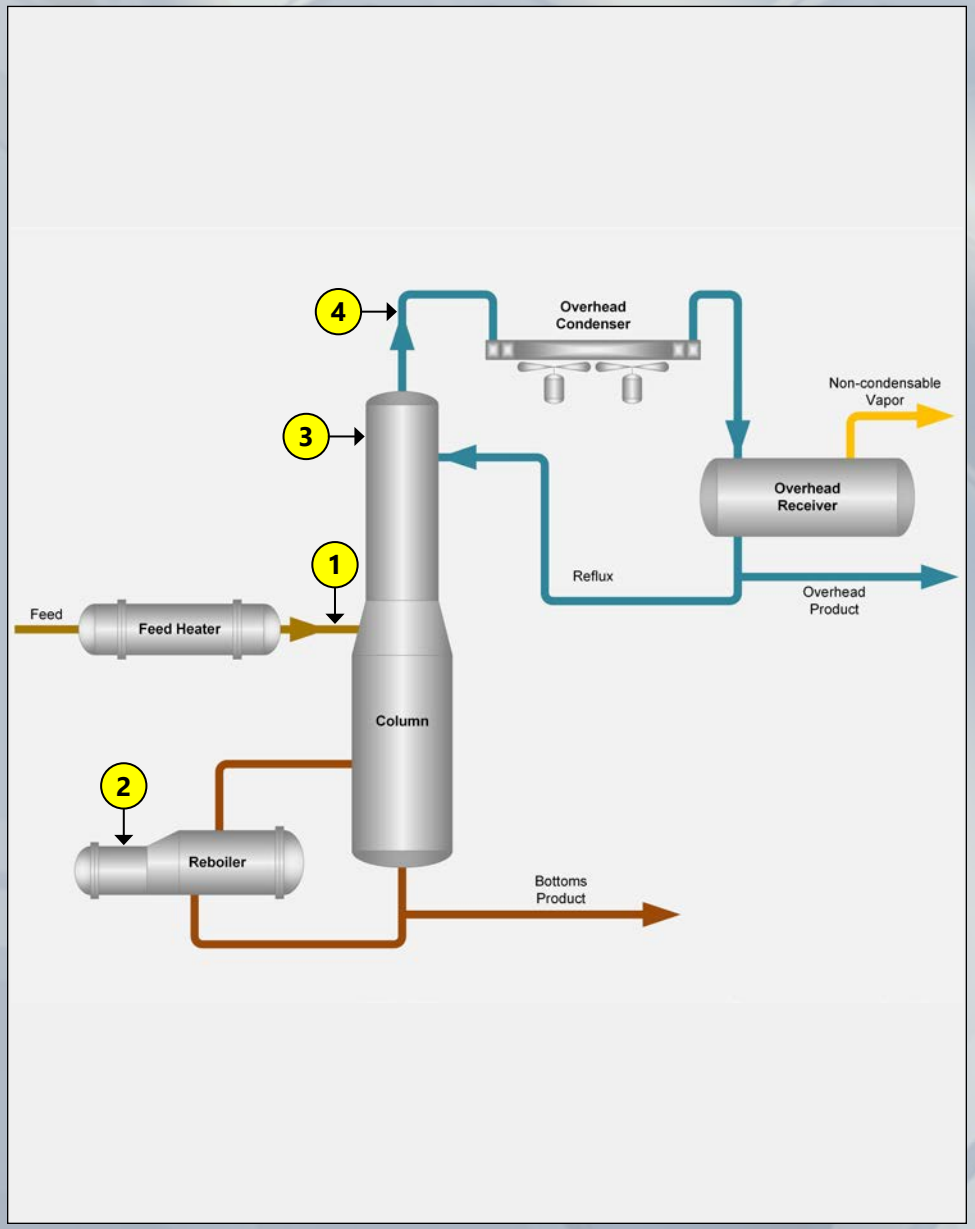
The graphic shows a typical **continuous distillation system**:

1. The feed mixture is continuously preheated and flows into the distillation column. Heating the feed supports vaporizing the more volatile component.

2. Liquid accumulates in the bottom of the column. It circulates through the **reboiler**. The reboiler is very important because ***it provides most of the heat required for distillation***. A reboiler can be compared to the Bunsen burner below the flask in the previous section. However, reboilers can be designed very differently as they may use steam, hot oil, or they can be a fired heater. It is important to know that the reboiler vaporizes the bottoms liquid and returns the vapor to the distillation column.

3. In the column, this vapor rises. It contacts falling liquid that has condensed from the feed and reflux. This vapor-liquid contact causes heat exchange between the hot rising vapor and the cooler falling liquid. As a result, mass transfer occurs between the vapor and liquid. There is continuous vaporization and condensation. Specifically designed column internals intensify the contact between vapor and liquid.

4. Vapor that has not been condensed by falling liquid exits the top of the column. This vapor is condensed by an **Overhead Condenser**.



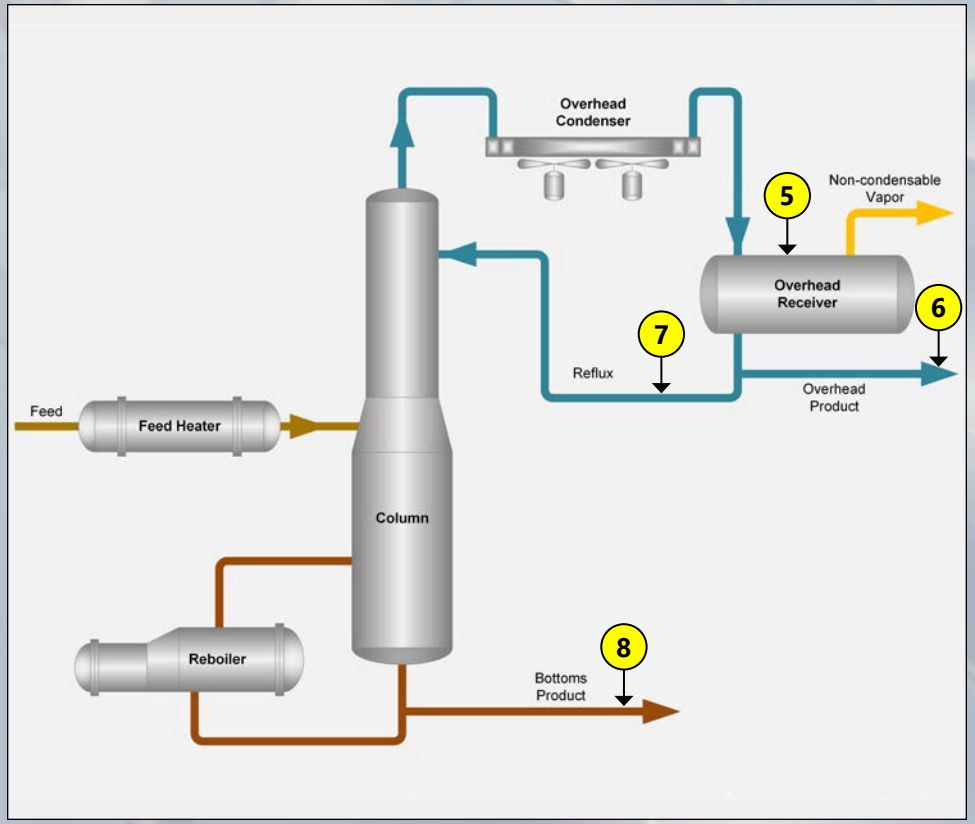
Equipment and Function (cont.)

5. The condensed liquid is collected in an **Overhead Receiver**. Any non-condensable vapor is vented from the receiver.

6. The overhead product is drawn from the overhead receiver. Overhead product is ***the more volatile component contained in the original mixture.***

7. A portion of the condensed liquid returns to the top of the column as **reflux**. Reflux is important because it provides cool liquid to the column and participates in vapor-liquid contact.

8. Bottom product, ***the less volatile component***, is continuously drawn from the bottom as a liquid.



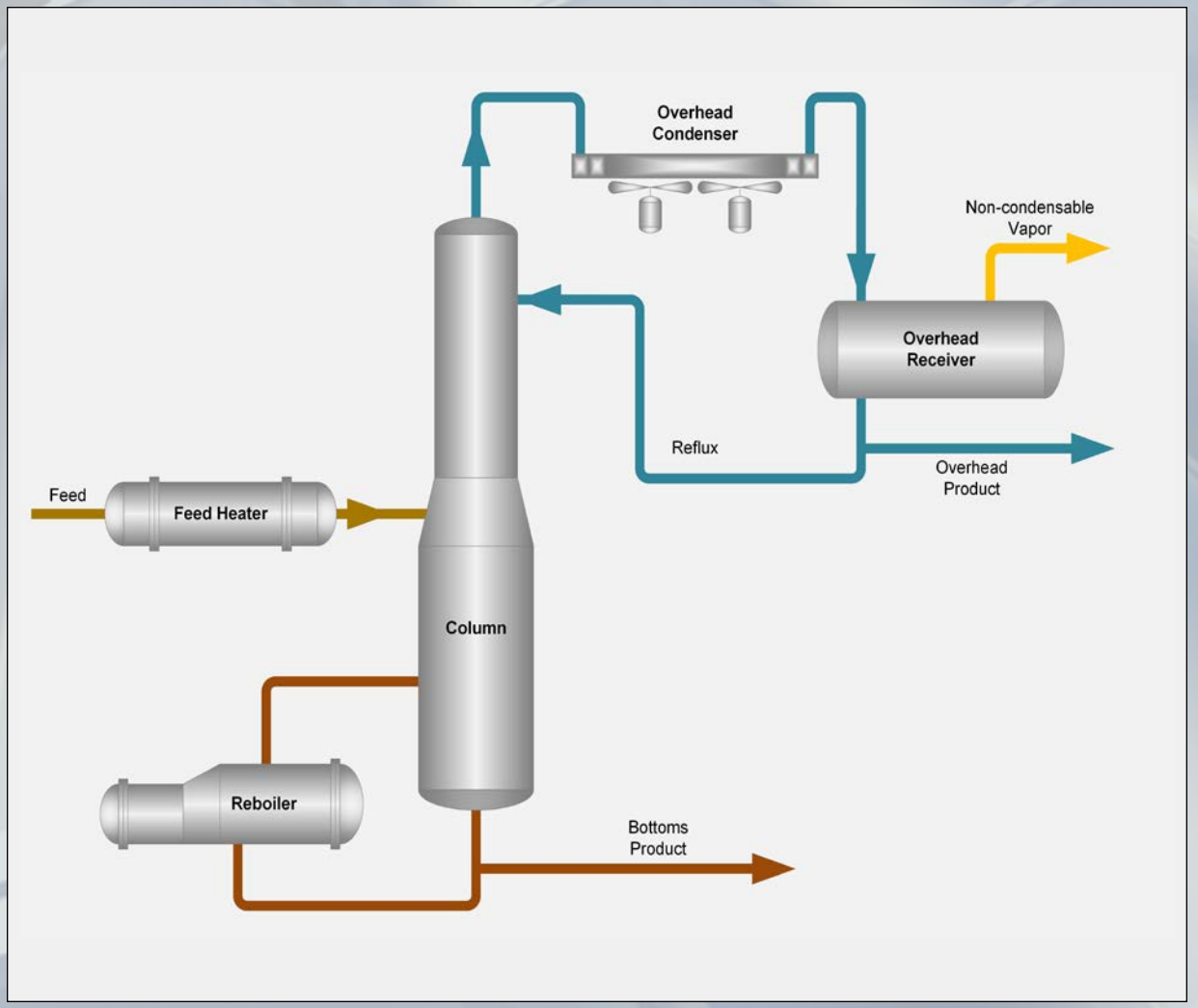
Typical Products

Based on the process, one or more streams are desired products. In many distillations, the overhead product is the more valuable product.

Products of Distillation

The products of distillation typically include:

- ***Overhead product*** containing the more volatile component. If hydrocarbons are distilled, the stream is called the lighter stream.
- ***Bottom product*** containing the less volatile components. If hydrocarbons are distilled, the stream is called the heavier stream.
- ***Non-condensables*** can be viewed as a third product. If hydrocarbons are distilled, the stream can be used as fuel gas.
- Sometimes a column has ***side draws*** (not shown). If a mixture contains more than two components, additional products can be separated and drawn along the column.



Overhead System

The term overhead system within a distillation process refers to all equipment that is associated with the vapor that exits the top of the column, the reflux, and the products that are retrieved from this vapor.

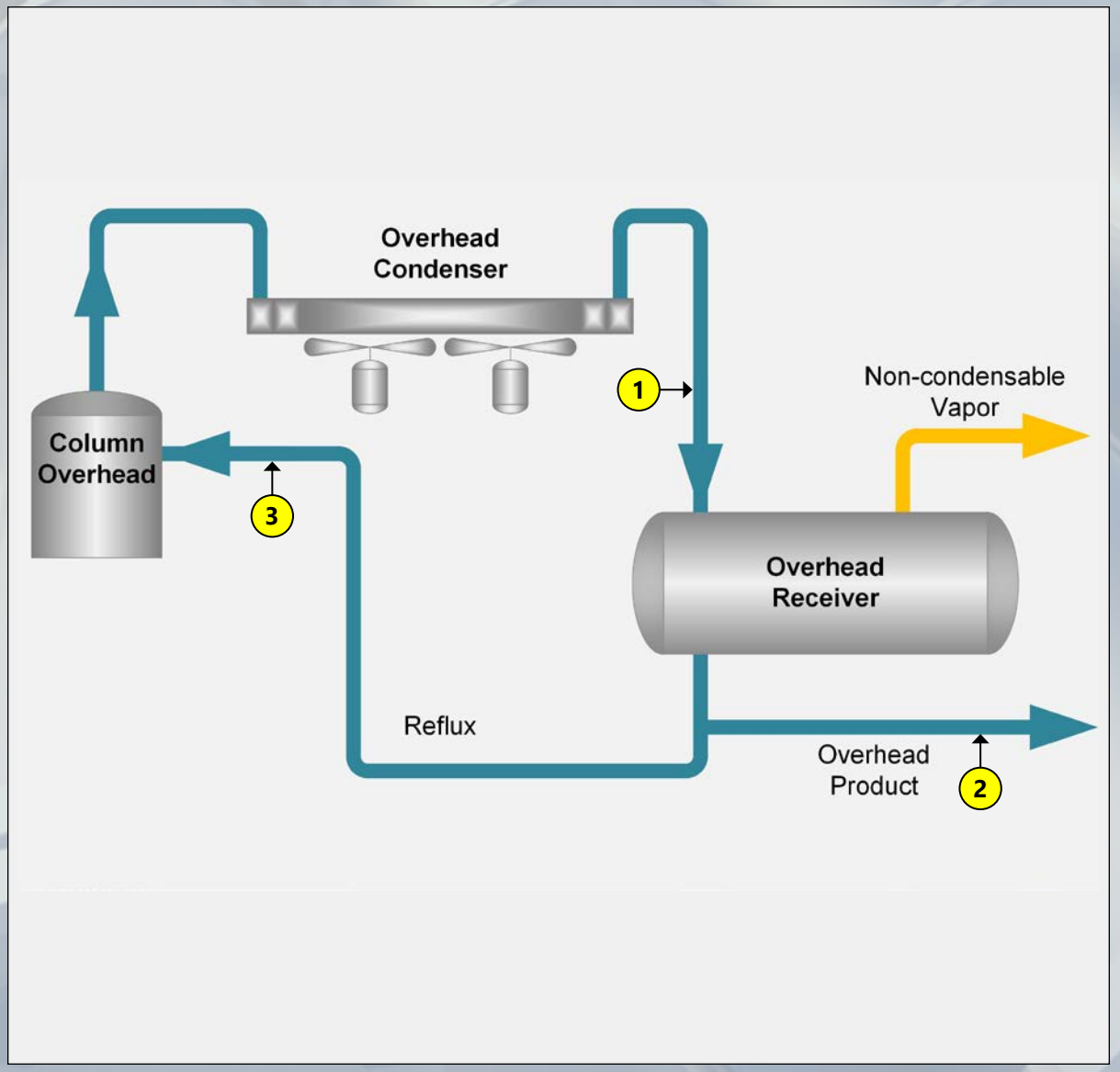
The **Overhead System** receives the vapor stream (the most volatile components) from the distillation column. The vapor temperature is close to the boiling point of the most volatile component contained in the column's feed.

1. The **Overhead Condenser** removes heat from the vapor, thereby condensing the vapor. The condensed liquid is collected in the **Overhead Receiver**. The receiver also operates as a separator. If some vapor remains uncondensed, it will be separated in the receiver.

2. The liquid in the receiver is the overhead product.

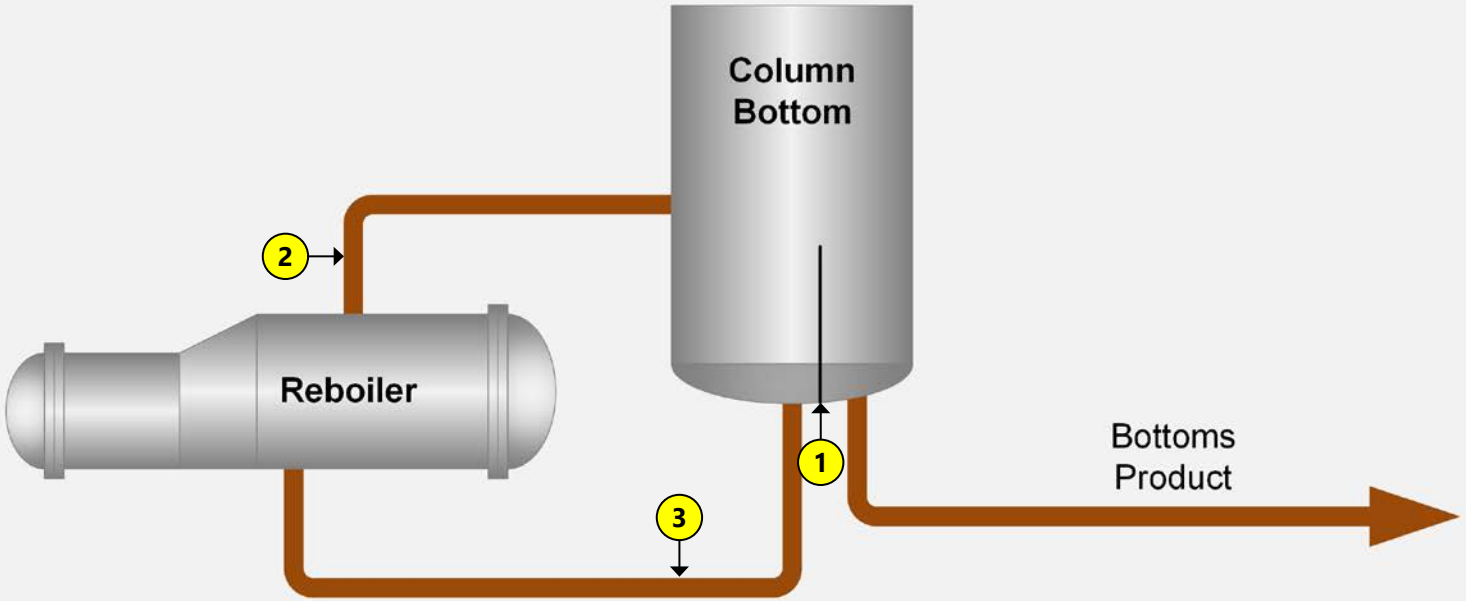
3. A portion of this cooled liquid returns as **reflux** to the column. Reflux has the same composition and temperature as the condensed overhead product. It provides cooling and condensing for the column.

The overhead product is often the desired product in distillation. The intention is to optimize purity of this stream. Reflux supports this because it condenses heavier vaporized material that has reached the Column Overhead.



Bottoms System

The term bottoms system of a distillation refers to all equipment that is associated with the liquid that accumulates in the bottom the column, the reboiler, the vapor that the reboiler generates, and the bottom product.



The **Bottoms System** collects the less volatile liquid from the distillation column. The temperature in the bottom is close to the boiling point of the less volatile component contained in the column's feed.

1. Sometimes, the column bottoms is separated into two compartments. One compartment is used to draw the bottoms product, and the other is used to supply bottoms product to the Reboiler.

2. The **Reboiler** drives distillation by providing heat to the column. Bottoms product partially or totally vaporizes in the Reboiler and returns to the column below all trays/packings. ***The amount of heat supplied has a strong effect on distillation.*** Most of the heat is supplied by the reboiler, but the preheated feed also supplies some heat to the column.

3. The liquid from the column to the Reboiler may be pumped or flow without a pump by employing the principle of a **thermosiphon.**

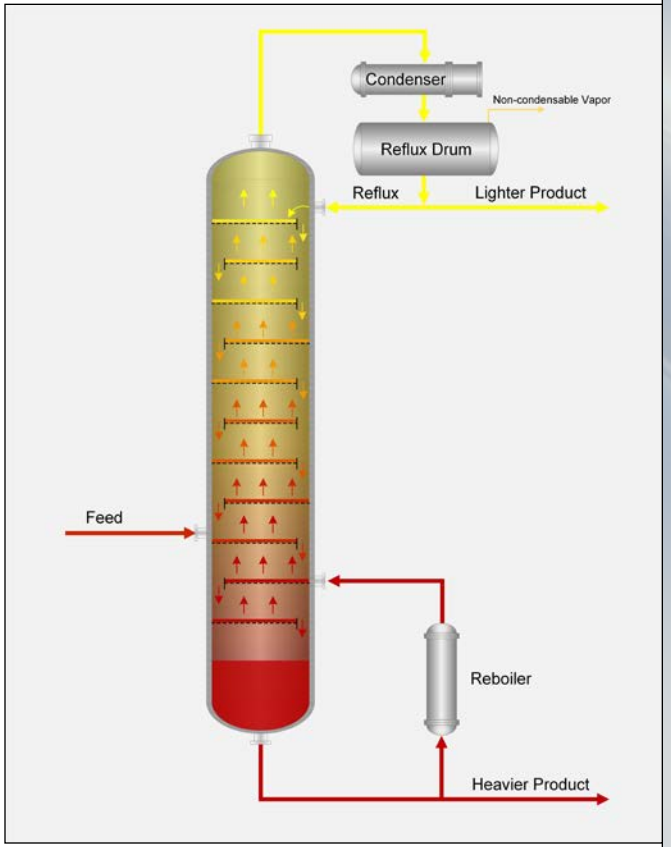
Material Balance

The material balance looks at streams coming into a column and flowing out of a column. When these are equal, the material balance for a distillation column is maintained. The material balance must be maintained to ensure continuous distillation.

Simply stated, ***the feed rate into the column must be equal to the total flow rates of overhead and bottom products***. The column always holds an inventory of material.

The following statements are true:

- If the feed rate ***increases***, at least one of the product streams must ***increase*** and vice versa.
- If the feed rate is constant and the flow rate of bottoms product ***increases***, the flow rate of overhead product ***decreases***. If the flow rate of overhead product ***increases*** at constant feed rate, the bottoms flow rate ***decreases***.
- If the feed rate is constant and one or more product flows ***decrease***, material accumulates in the column (the level ***increases***).
- If the feed rate is constant and one or more product flows ***increase***, material depletes in the column (the level ***decreases***).
- If the feed rate ***increases*** and the flow rate of both product streams is constant, material accumulates in the column (the level ***increases***).



It is important to note that the reboiler circulation and reflux circulation do not need to be considered for the overall column material balance. They are internal streams that do not affect the material balance However, they can shift the material balance ***indirectly*** by changing the temperatures as described on the next page.

Reflux Flow is used to calculate a **Reflux Ratio**. ***The higher the reflux ratio, the purer the overhead product.*** It is calculated by dividing the reflux flow rate by the overhead product flow rate(s):

$$\text{Reflux Ratio} = \frac{\text{Reflux Flow}}{\text{Flow of Liquid + Non-condensables}}$$

Heat Balance

The heat balance looks at heat supplied to a column and heat removed from a column. When these heat streams are equal, the heat balance for a distillation column is maintained. The heat balance must be maintained to ensure continuous distillation.

In continuous distillation, the objective is to maintain steady state operation. **Steady state operation** means that the temperatures, pressure, flow rates, and composition ***do not change over time***. Because heating and cooling are the main drivers to establish temperatures, heating and cooling for a column must be balanced.

In order to maintain constant temperatures along the column, the following must be accomplished:

Heat In = Heat Out

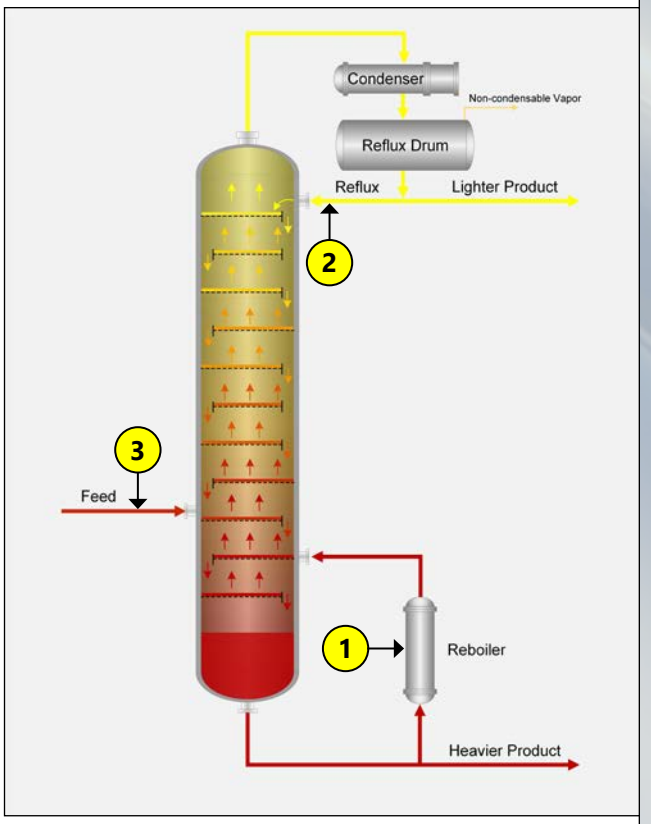
Heat is supplied by the reboiler and the preheated column feed.

Cooling is provided by reflux. If the feed is cooler than the tray at which it enters the column, it is also a source of cooling.

The following describes changes that will occur if heating or cooling changes:

1. If the reboiler supplies ***less heat than required***, the column will cool down and less vapor will rise through the column. As a result, distillation can be jeopardized and the more volatile component may not be vaporized.
   If the reboiler provides ***too much heat***, more of the heavier component will vaporize and may reach the overhead vapor.

2. If ***reflux flow is too low***, temperatures will increase and less of the heavier component that reaches the upper column will be condensed. As a result, it can reach the overhead and accumulate in the overhead product. The overhead product may be off-spec and bottoms product will be lost to the overhead product.



3. If the ***feed is too cool*** or the ***feed rate increases***, the column will cool down. As a result, more liquid will fall to the bottom and the more volatile component will be condensed.

In conclusion, it is necessary to adjust heating when cooling changes (and vice versa) to maintain the desired column temperatures and product composition.

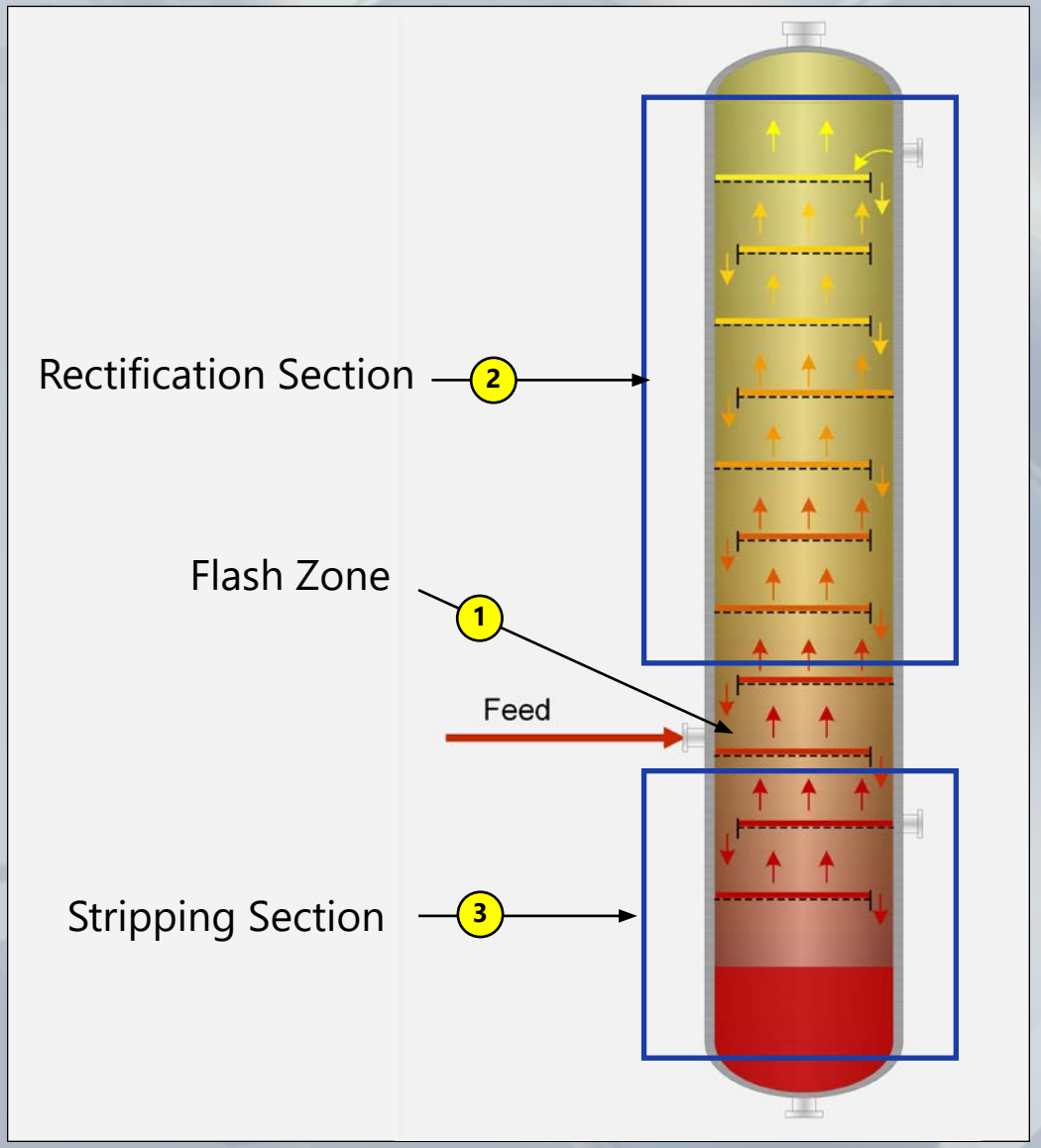
The Distillation Column

The column, the heart of a distillation system, is shaped like a vertical cylinder. Columns vary in height and diameter and may have specific internals, but some features are common to all distillation columns.

1. The area where feed enters the column is called the **flash zone**. Because the feed is typically preheated, some flashing occurs. The vaporized portion of the feed rises and the liquid portion falls toward the bottom.

2. The **rectification section** is the part of the column above the flash zone. Here, ***the concentration of the more volatile component is high***. Rectification refers to the fact that any less volatile (heavier) material is condensed by reflux liquid. The condensed, less volatile portion falls toward the bottom.

3. The **stripping section** is located below the flash zone. Here the concentration of less volatile material is high. Stripping refers to the fact that vapor generated by the reboiler rises through the liquid and strips material by turning it into vapor. The vapor rises toward the top.



Distillation Trays

Distillation trays are the most common internals for distillation columns. Many trays are arranged horizontally along the height of the column. Distillation trays are necessary to perform multi-stage distillation in one column.

Distillation trays are widely used as internals in distillation columns. The purpose of trays is to intensify the contact between vapor and liquid. Trays can be designed differently, but their basic function is similar. The principle is described below:

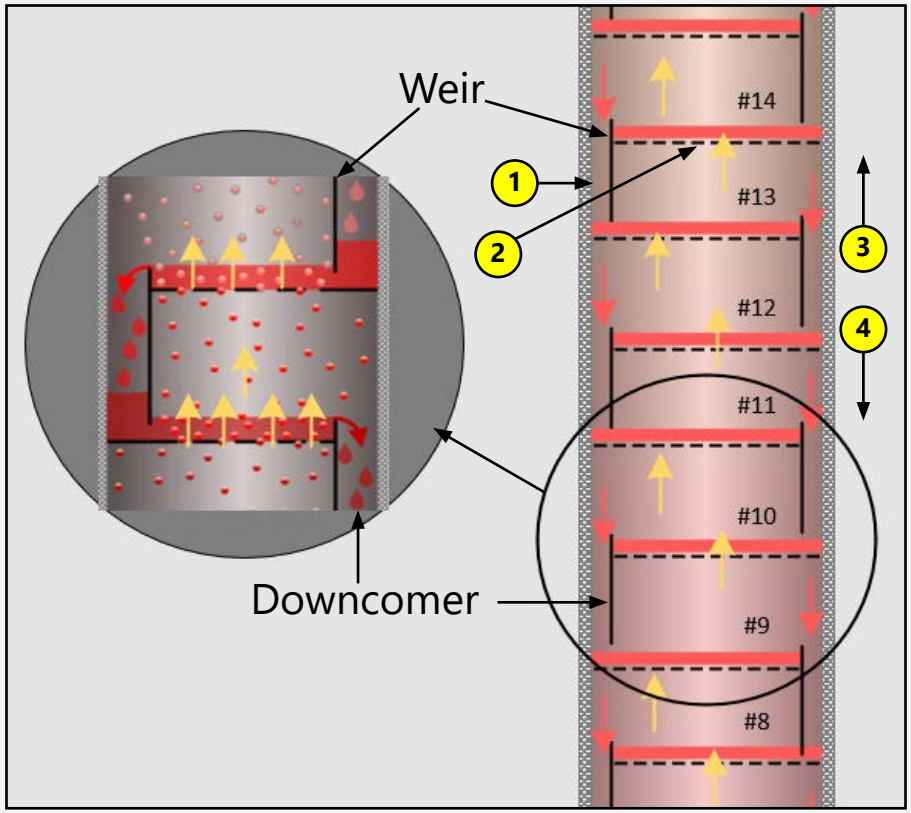
1. Liquid that falls through the column accumulates on each tray. It forms a level and overflows a **weir**. It then falls through a **downcomer** onto the next tray.

2. The trays have small openings. Vapor rises through the openings and bubbles through the liquid on each tray. ***This is where heat exchange and mass transfer between hot vapor and cooler liquid occurs.*** The hot vapor vaporizes the volatile components contained in the liquid. At the same time, the cooler liquid condenses the less volatile components contained in the vapor.

3. Because vapor rises from tray to tray, the more volatile component becomes purer towards the top. Trays higher up in the column have a lighter composition.

4. The less volatile component becomes purer towards the bottom. Trays further down have a heavier composition.

Each tray can be viewed as a single stage distillation. Each tray is at equilibrium; on each tray, the temperature and composition of the vapor and of the liquid remain constant. However, ***on each tray, the composition of vapor is different from the liquid composition***. The vapor always contains more of the volatile component than of the less volatile component. The composition changes from tray to tray.



Equilibrium in a Distillation Column

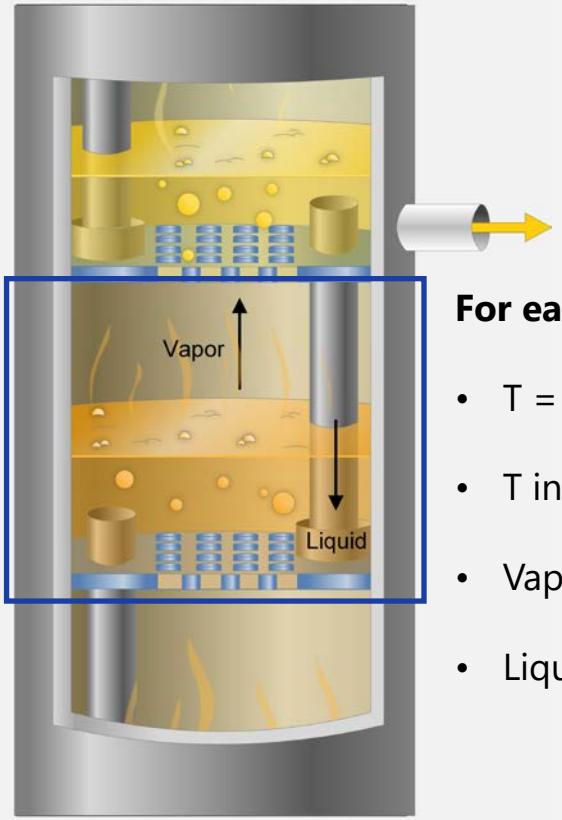
Vapor-liquid equilibrium exists on each tray in a continuously operated distillation column. Each tray is similar to a single stage distillation.

Recall that equilibrium in distillation means: ***the amount of vaporization is the same as the amount of condensation***. The liquid composition and the vapor composition do not change over time.

That being said, the liquid composition and the vapor composition are not the same. Each tray in a continuous distillation column is at equilibrium.

Each tray holds liquid of a specific composition. Its temperature is this specific mixture's boiling point. The vapor above the liquid is at the same temperature.

The composition of the vapor is different from composition of the liquid. The vapor contains more of the more volatile component, and the liquid contains more of the less volatile component. Composition and temperature do not change over time.



For each tray:

- T = constant over time
- T in Liquid equals T in Vapor
- Vapor Composition is lighter than Liquid
- Liquid Composition is heavier than Vapor

Equilibrium on Trays

Pressure-Temperature Profile in a Distillation Column

Pressure and temperature in a distillation column do not have equal values along the height of a column. Instead, there is a temperature profile and a pressure profile.

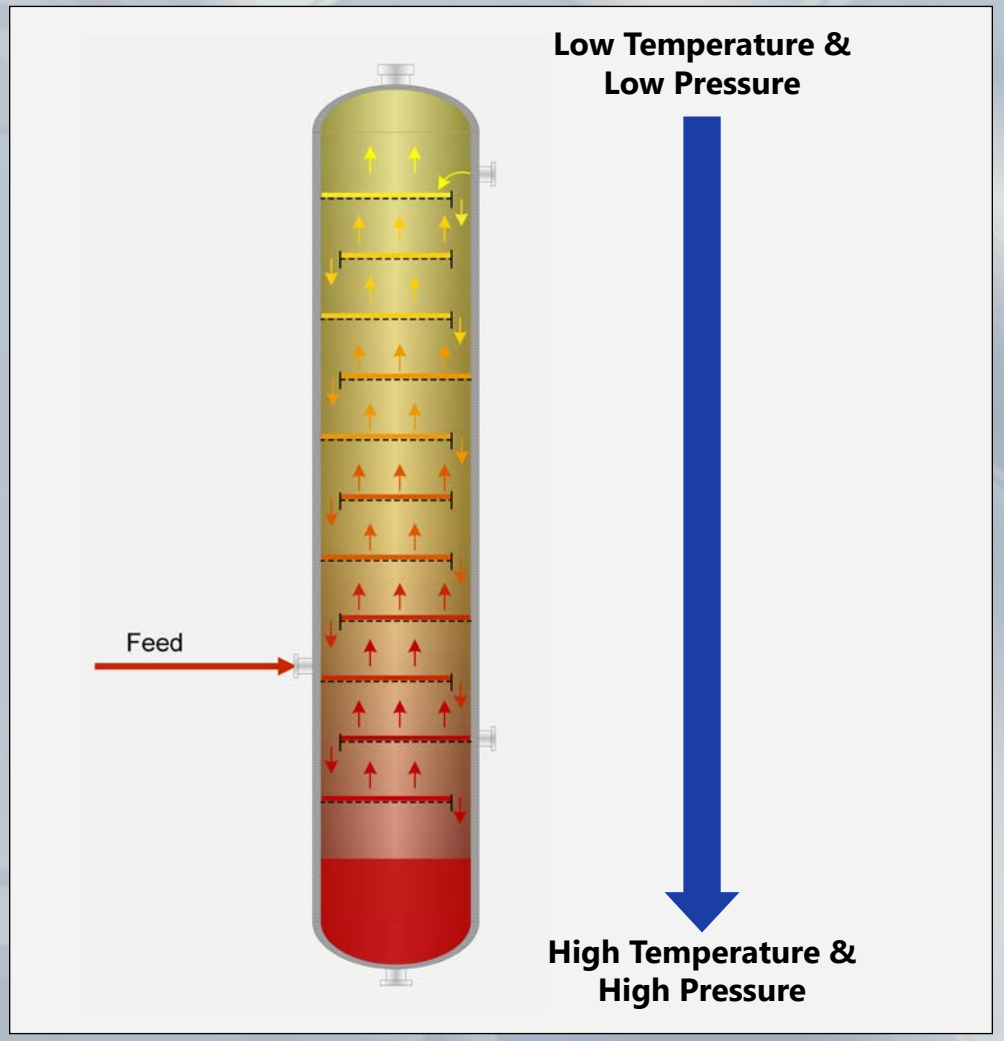
The temperature in a distillation column changes along the height of the column. The highest temperature is near the bottom, and the lowest temperature is near the top. ***The temperature decreases successively between trays from bottom to top.***

This can be easily remembered by looking at where heating and cooling are provided: **reflux cools, the reboiler heats.**

The temperature changes along the column because each tray holds slightly different composition of the liquid mixture. The change in composition causes the mixture to boil at different temperatures.

Higher temperatures are associated with ***heavier*** material; lower temperatures with ***lighter*** material. The top tray boils at nearly the boiling point of the lightest, most volatile material. The bottom tray boils at nearly the boiling point of the heaviest, less volatile material.

Pressure in the column also changes in this direction.



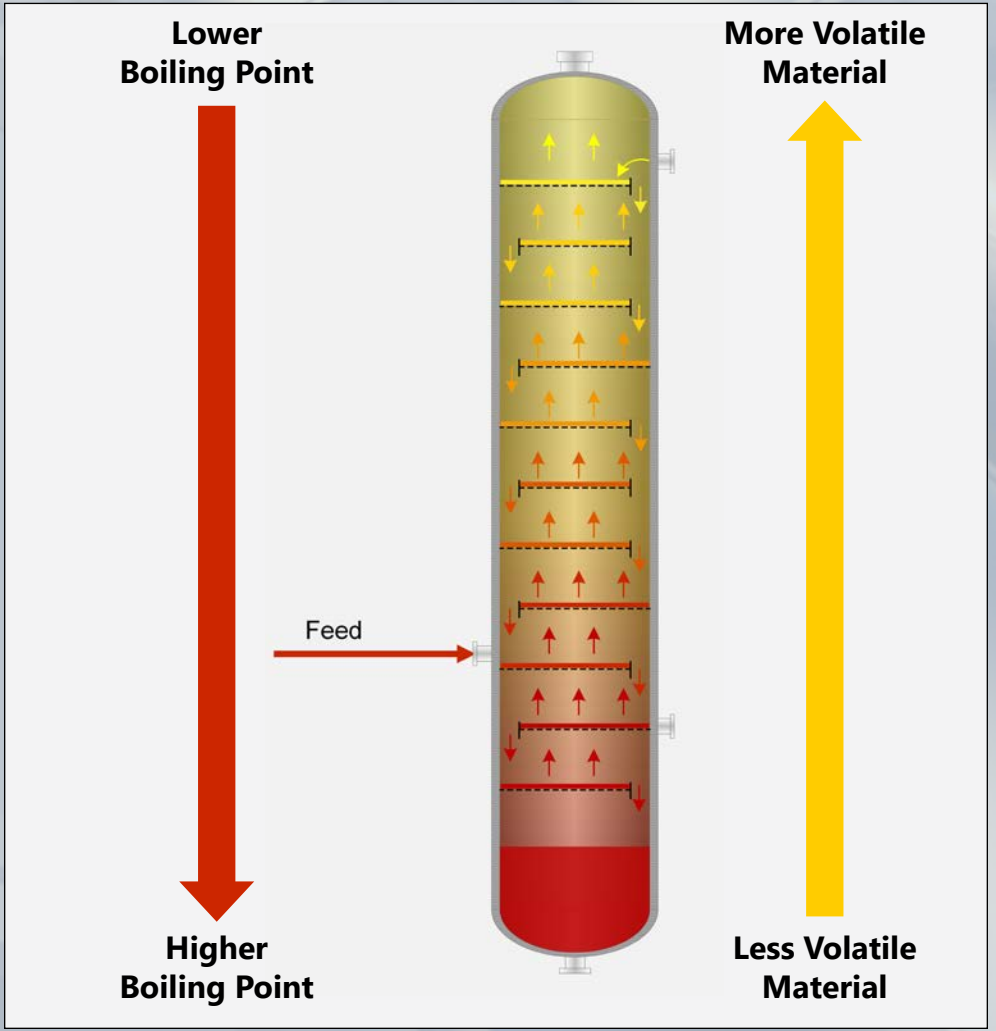
Composition Profile in a Distillation Column

The composition in a distillation column changes along the height of a column. This fact allows you to draw the more volatile product from the top of the column and the less volatile component from the bottom of the column.

The composition of a boiling mixture changes with temperature. All trays contain boiling liquid, but the temperature decreases on trays upward the column. ***Due to the continuous mass transfer, the composition on trays is lighter further up in the column, and heavier further down the column.*** As such, the light material is drawn from the column top, and the heavy material is drawn from the column bottom.

Each tray has its own specific vapor/liquid composition that does not change, as long as heating or cooling at the column remains constant.

For a specific distillation, it is therefore possible to know the composition on each tray, top, and bottom by looking at temperatures.



Column Internals

Distillation columns have internals that promote distillation. Most commonly, these are trays. Packings are also used, and some columns have a combination of trays and packings. There are various types of trays, each with different features.

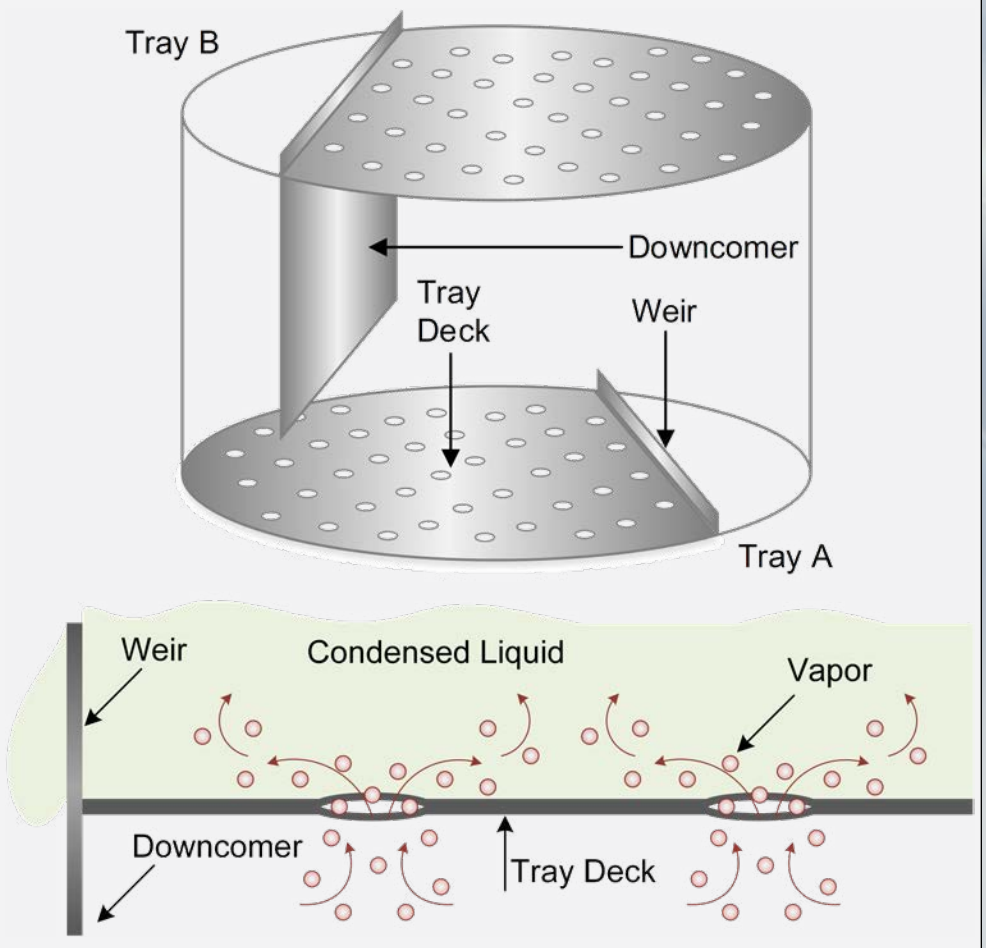
Sieve Trays

Sieve trays are perforated metal plates.

Hot vapors travel upward through the perforated holes in the tray deck, bubbling through the condensed liquid present on the tray.

The pressure of the vapor traveling up through the holes exceeds the pressure exerted on the tray deck by the liquid. This prevents the liquid from flowing down through the holes in the deck so the liquid must flow over the weir and down the downcomer.

Sieve trays are cheaper than other trays, but do not efficiently adapt to load changes in the column.



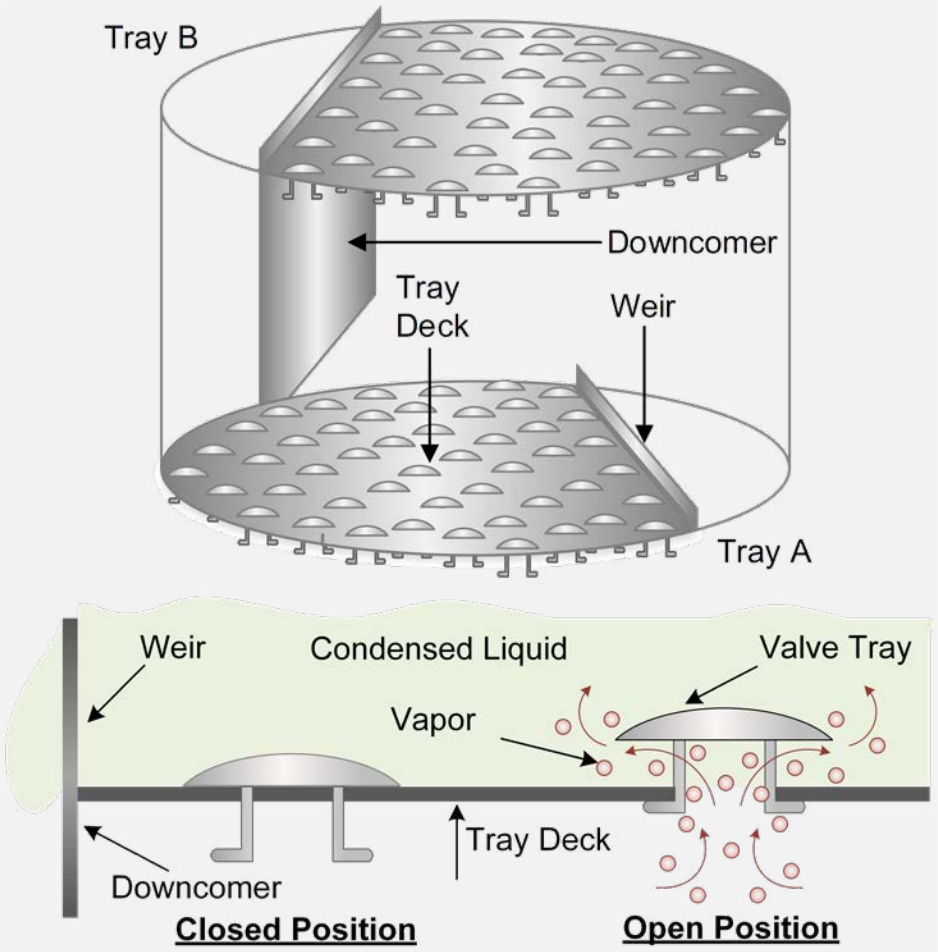
Sieve Tray

Valve Trays

Valve trays are equipped with covers over the perforations. These covers move up and down, based on the pressure exerted by the rising vapor.

Hot vapors travel upward, pushing against the bottom of the tray deck. The pressure of the vapor lifts the valve caps. The vapor exits through the sides of the cap, rising through the condensed liquid on the tray. The liquid level presses down on the valve cap and prevents the liquid from flowing down through the openings, forcing the liquid to flow over the weir and down the downcomer.

Valve trays are more expensive, but offer a wider operating range with regard to column load.



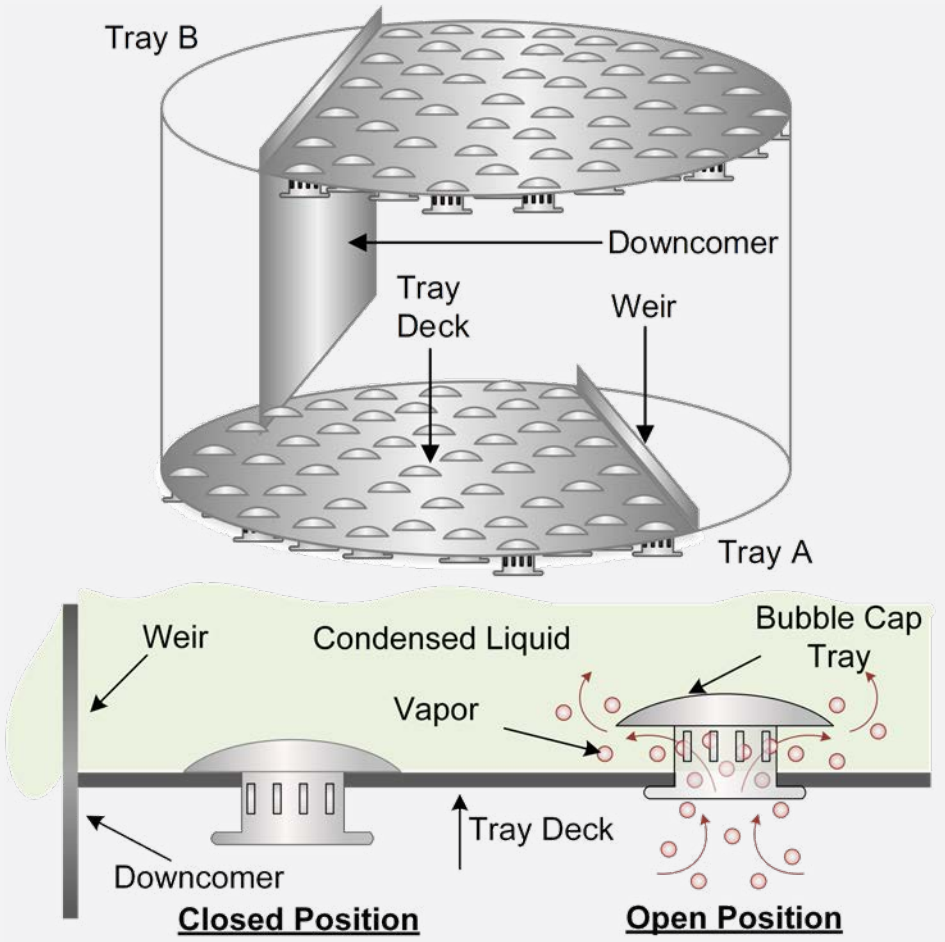
Valve Tray

Bubble Cap Trays

Bubble cap trays are similar to valve trays, but the moving caps are equipped with smaller openings.

Hot vapors travel upward, pushing against the bottom of the tray deck. The pressure of the vapor lifts the bubble caps. The vapor exits through the grids in the cap, creating smaller vapor bubbles. It rises through the condensed liquid collected on the tray. The liquid level presses down on the cap and prevents the liquid from flowing down through the grids in the deck, forcing the liquid to flow over the weir and down the downcomer.

Bubble cap trays are more expensive, but offer the widest operating range for load changes.



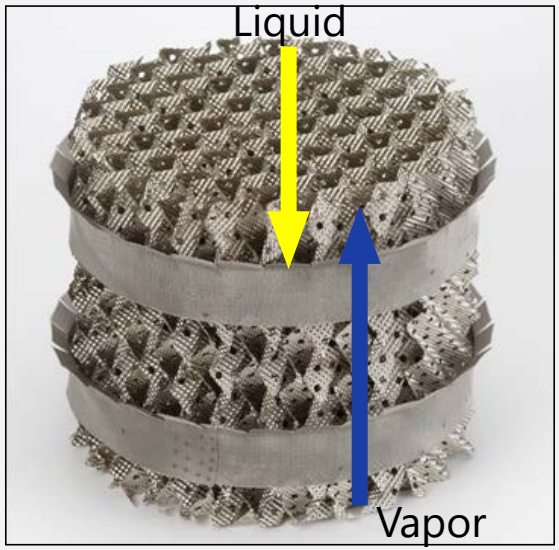
Bubble Cap Tray

Other Internals

Packings are often used instead of trays. Some columns have a combination of packings and trays. Packings are tightly packed perforated metal sheets. Like trays, they intensify the vapor-liquid contact, but they do not hold a liquid level.

The benefit of packings is the much lower differential pressure along the column compared to trays. The reason for this is that there are no internal liquid levels. Lower differential pressure is beneficial for the hydraulics (vapor-liquid traffic) in the column.

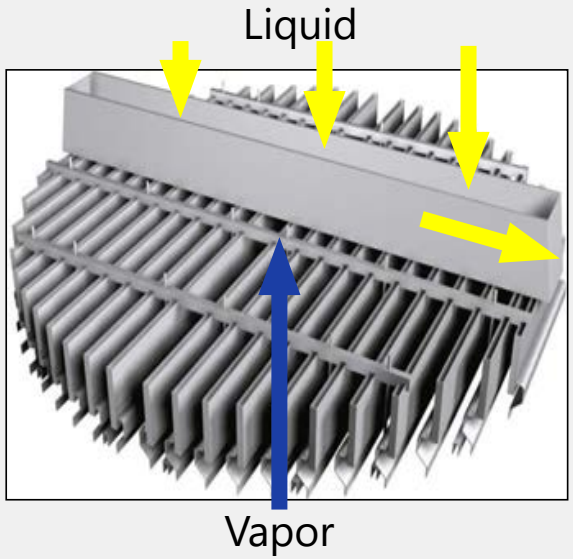
However, packings are more expensive than trays and can not be cleaned mechanically.



While all other trays described ensure intimate mixing of vapor and liquid, the **knockout tray** is specifically designed to keep vapor and liquid separate.

This tray is often in place to collect large amounts of liquid to draw-off a product. It is also used to collect liquid for altering the distribution to column trays/packings below. Sometimes, they are called **chimney trays.**

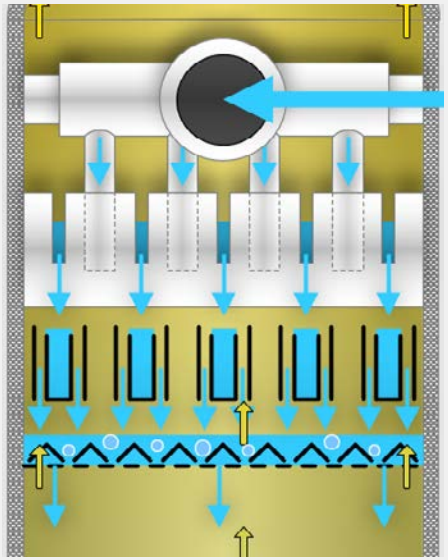
The knockout/chimney tray is also designed to provide good vapor distribution to trays or packed sections above it.



Distributors in columns are in place to ensure even flow of feed and reflux across the column diameter. They can distribute liquid, vapor, or both.

Distributors are specifically designed for the expected flow rate. They direct the flow over the desired area and create the smallest possible pressure drop when the fluid flows through the distributor.

The graphic below shows an example of a liquid distributor inside a column. It is connected to the inlet flange. A set of weirs is arranged across the diameter below it. Liquid overflows these weirs, and is evenly distributed as it falls downward.



Fractionation Systems

Fractionation is the process of separating a mixture into fractions. A fraction is a portion of a mixture that has specific properties. Previously discussed distillation systems separate a mixture into two fractions: an overhead and a bottom fraction. In the industry, the term fractionation is used to label columns that separate a mixture into more than an overhead and a bottom product.

Purpose and Function

Fractionation is a complex distillation method that separates multi-component mixtures into more than just an overhead and a bottom product. Mixtures that contain multiple components, such as crude oil, can be separated into ***several product streams*** using fractionation.

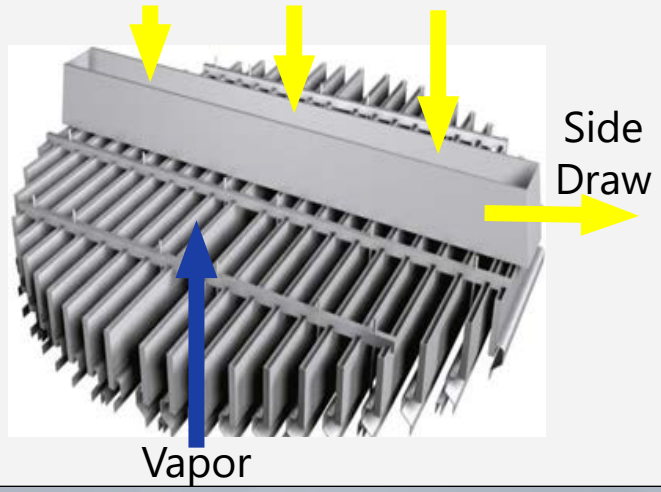
This is accomplished by drawing product streams at different heights along the fractionation column. The product streams are also called **fractions** or **cuts**.

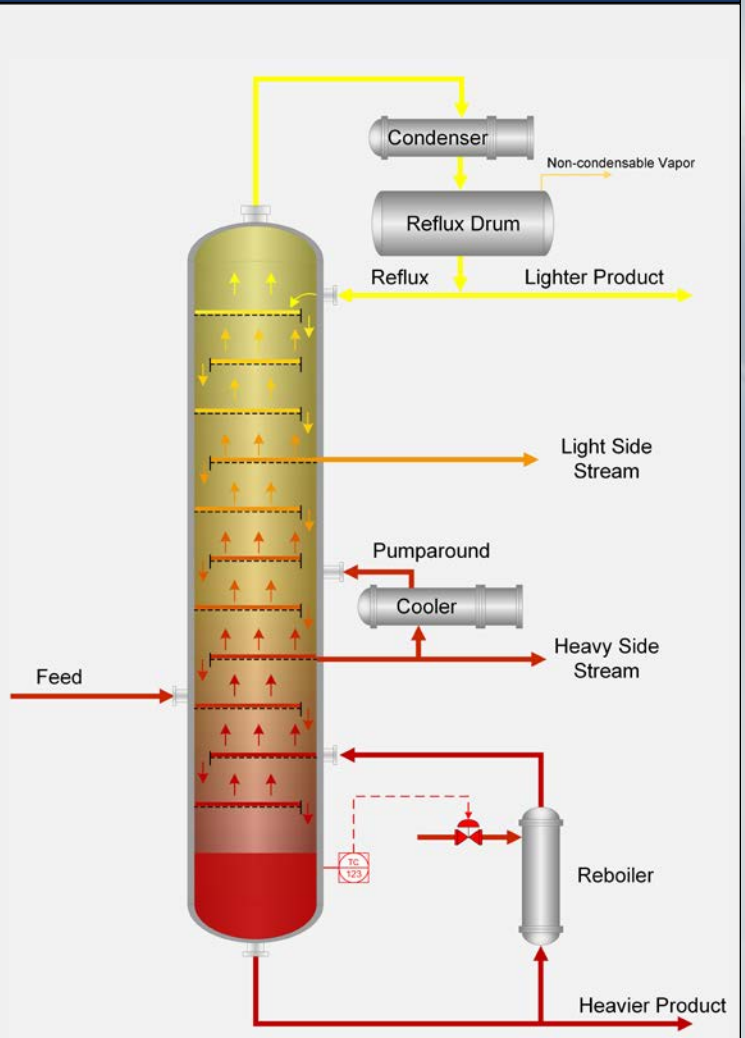
You have already learned that different components have different boiling points and that each tray in a tower is at a different temperature/composition. **Side stream products** are drawn from the trays where the desired component's concentration in the boiling liquid is the ***highest***, compared to other trays.

Similar to distillation systems, fractionation systems are equipped with reboilers and overhead condensers. Sometimes, reflux from the drawn side streams is circulated back to the column above or below the associated draw tray; these streams are sometimes called **pumparounds**.

For a heat and material balance of a fractionation column, the side streams must be included.

The draw trays are specifically designed to enable product draw off. This can be, for example, a knockout tray as shown on the right.

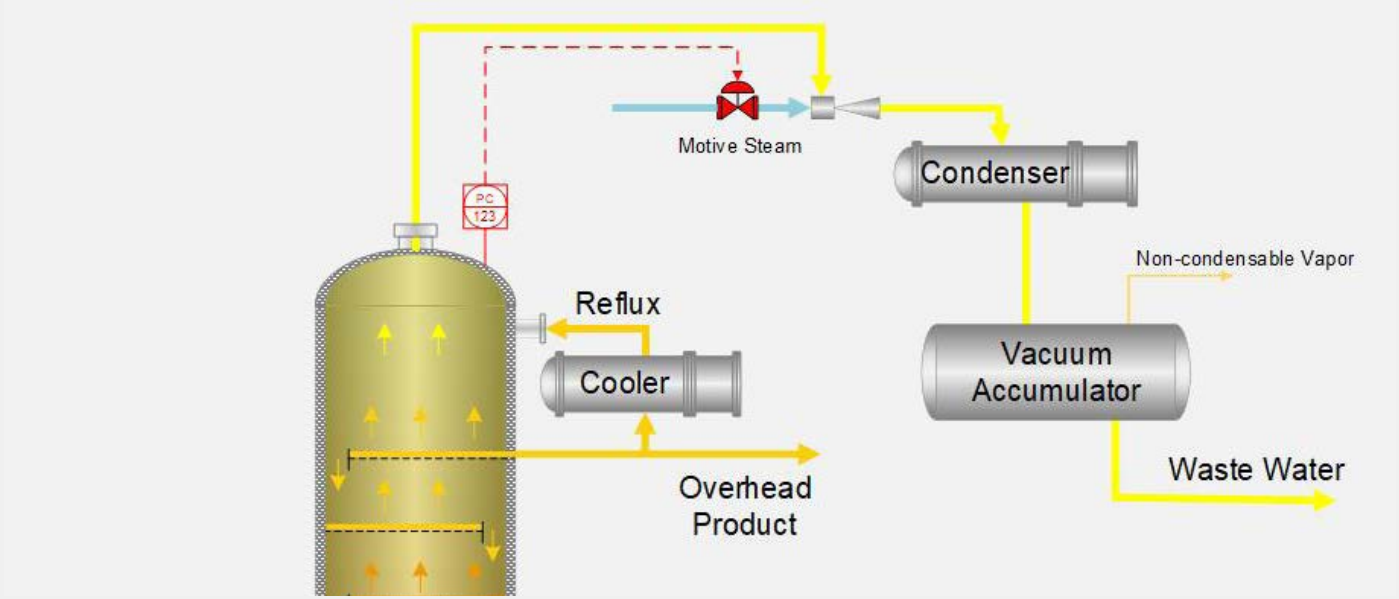




Vacuum Distillation

Some distillation columns operate under **vacuum**: a pressure below atmospheric pressure. A vacuum system, attached to the top outlet of the distillation column, creates the low pressure in the column.

Purpose



Recall that lower pressure reduces the boiling points of components and mixtures. This physical relationship is exploited by distilling a mixture under vacuum: ***The lower pressure allows for operating the column at lower temperature***.

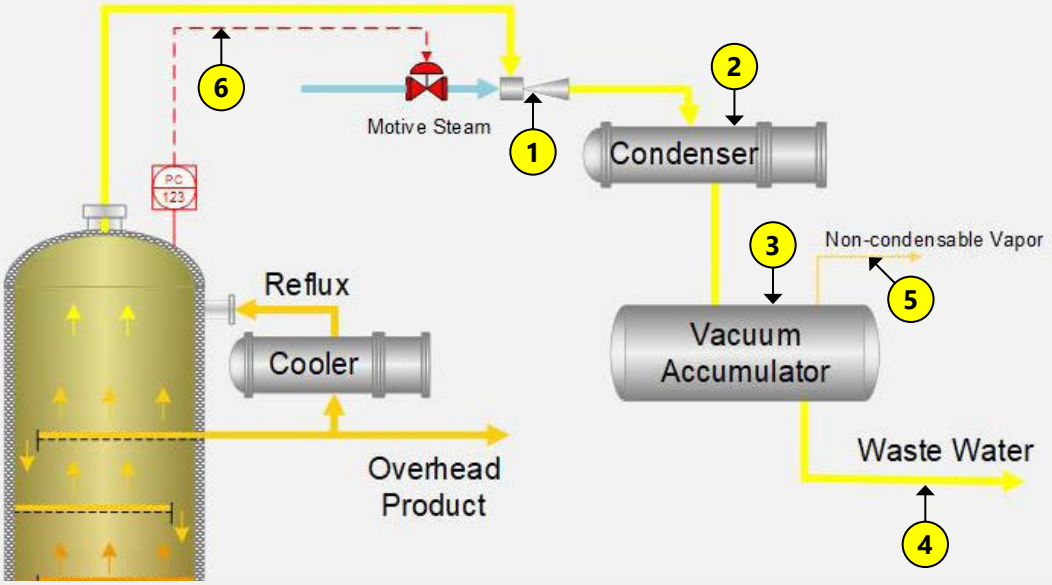
This can be beneficial for the following reasons:

- Products that are sensitive to higher temperatures can be distilled at lower temperatures
- Some compounds can cause coking and deposits at high temperatures in the column

The vacuum column operates in the same manner as a typical distillation column. The low pressure is created by a **vacuum system**, located in the overhead section of the distillation system.

The vacuum system continuously draws vapor containing inerts and light ends from the column, thereby creating a low pressure in the column. This stream is not considered being an overhead product.

Function



The vacuum system operates as follows:

1. An **ejector**, or a set of ejectors, draw vapor from the top of the column. Ejectors use steam as a motive fluid to draw suction on the vapor.

2. Steam and overhead vapor combine in the ejector. The combined stream is exhausted into the **Overhead Condenser**. Here, cooling water condenses the combined stream.

3. The condensed stream flows into the **Vacuum Accumulator**. The inlet pipe routes the combined stream below the liquid level in the accumulator. This creates a seal so that atmospheric air does not enter into the drain pipe and flow back into the column.

4. The accumulated liquid is a mixture of condensed steam and the drawn overhead components. From here, it is typically sent to a water treatment unit.

5. Uncondensed vapors are vented to the atmosphere. These are inerts drawn from the column that cannot be condensed with cooling water. There may be some residual steam as well.

6. The overhead pressure of a vacuum column can be controlled by adjusting the amount of motive steam flow. More steam creates a lower pressure, and vice versa. Some units may control the pressure by adjusting cooling water flow through the condenser.

The lowest pressure can be measured at the top of the column. As with any other column, the pressure increases top to bottom. The lightest column product is drawn below the overhead vapor exit; there is no product drawn from the vacuum system.

Variables Affecting Distillation

Specific variables "drive" the distillation process. The Operator can adjust these variables by using controllers.

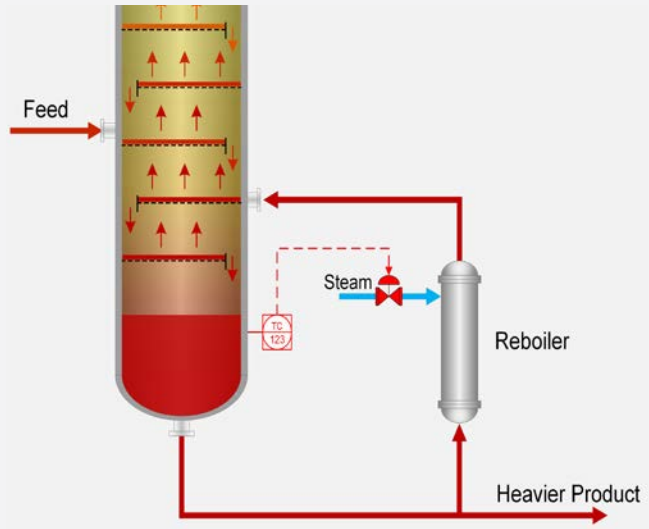
Reboiler Duty

Reboiler duty refers to the amount of heat supplied by the reboiler. More reboiler duty increases column temperatures, vaporizes more of the heavier material, and creates heavier column composition.

Too much reboiler duty may result in all products becoming too heavy. It can also create too much vapor, followed by hydraulic problems in the column.

Low reboiler duty may result in a cooler, lighter, and likely off-spec product composition. Increased amounts of the lighter component will fall to the bottom.

Oftentimes, the heat input by the reboiler is controlled by a temperature controller, located near the bottom of the column.



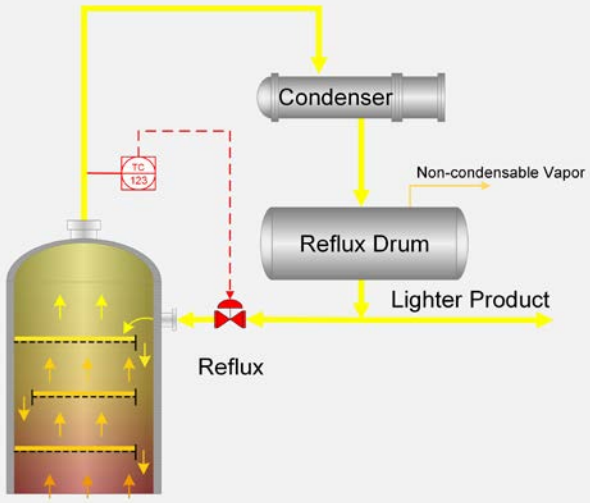
Reflux Flow

Reflux flow must be balanced with the heat input from the reboiler. If the reflux flow is ***too high***, the column temperatures will decrease, light material can condense, and the column composition will become lighter. As a result, all products may become too light.

In addition, high reflux flow can cause a high differential pressure along the column, followed by flooding.

Oftentimes, columns have a temperature controller near the top that adjusts the reflux flow to maintain a constant overhead temperature.

When balanced with heat input, ***increasing the reflux flow*** will increase the reflux ratio and improve the purity of the overhead product.

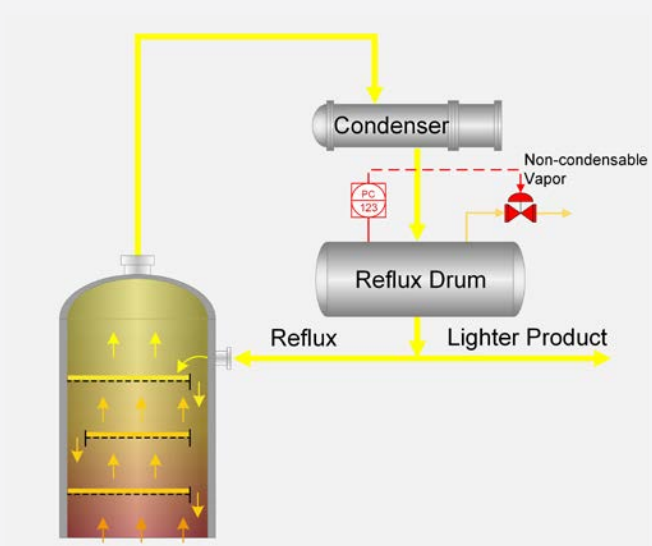


Pressure

Pressure directly affects boiling points. Because of this, pressure is controlled in almost all columns. The point of control is often in the ***Overhead Accumulator***. Controlling pressure here also supports condensation of the overhead vapor.

If the column pressure is ***too low***, the column may cool down and products may become lighter, and vice versa.

If the pressure is controlled (as shown in the graphic below), low pressure also means that less of the overhead vapor will be condensed and lost to the non-condensable vapor.



Composition

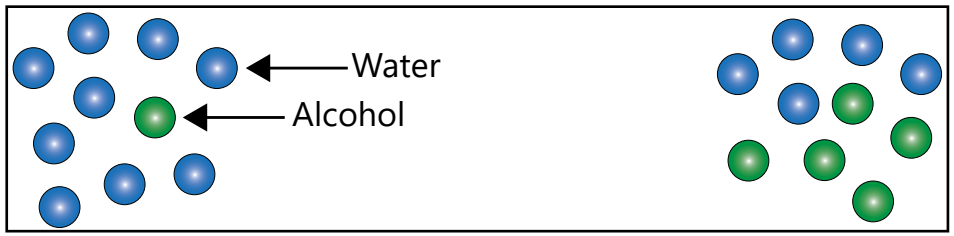
Composition is a term that describes how much of each component is in a mixture. There are several ways or units of measurement to express composition. These are used to quantify the composition of the feed mixture, and the composition of each distillation product. All measurements of compositions indicate concentration.

The **mole fraction** is one way to quantify the composition of the mixture. A mixture contains a total number of **moles** of all components. More specifically, the mole fraction is the number of moles of one component in proportion to the total number of moles in the mixture. The graphic below depicts two mixtures with different compositions:

The mole fraction of the more volatile component is high in the overhead product and low in the bottom product. The opposite is true for the less volatile component.

Note: Mole fractions are also used to quantify how much of a component on a tray or in a distillation stage is in the vapor phase and in the liquid phase.

Often, the concentration of a component is described in **percent (%)** as equivalent for mole fractions. For example, if the mole fraction is 0.1, the concentration of this component is 10%.



Total Moles: 10
Mole Fractions:
0.1 alcohol
0.9 water

Total Moles: 10
Mole Fractions:
0.5 alcohol
0.5 water

Mole Fractions in Mixtures

Parts per million (ppm) is used to quantify concentrations of very low percentage. 1 ppm is equal to 0.0001%.

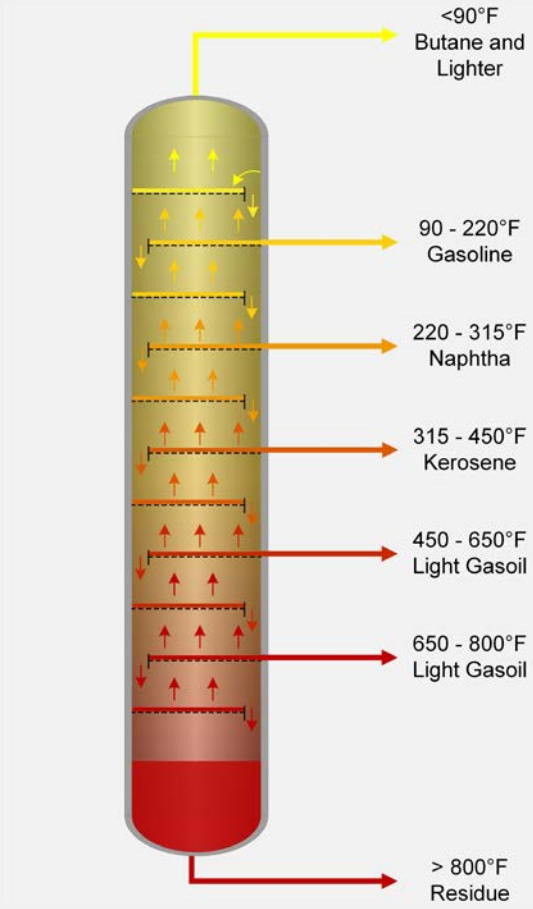
In distillation, it is desired to have a high concentration of the more volatile component in the overhead product while the concentration of the less volatile component must be low (often in ppm range).
In the bottom product, the concentration of the more volatile product must be low (often in ppm range).

Cut Points

The term **cut point** is often used in crude oil fractionation. It is the temperature at which a fraction can be separated from the heavier material of a mixture. Cut points for crude fractions are shown below.

The product temperature in a draw tray must be at the cut point for the component that is drawn from this tray.

In a fractionation column, the cut points increase from top to bottom of the column.

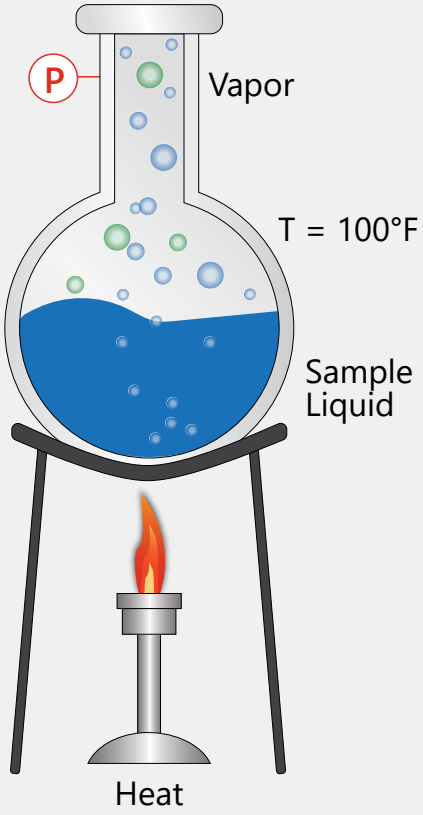


Reid Vapor Pressure

Reid Vapor Pressure (RVP) indicates the volatility of gasoline and other crude oil fractions. In the lab, a sample of the product is heated to 100°F in a closed flask. The vapor pressure at this temperature is measured.

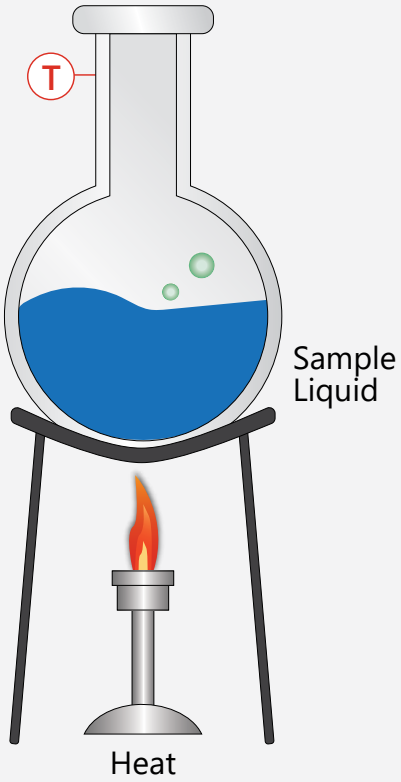
This total vapor pressure provides information about the concentration of components in the sample.

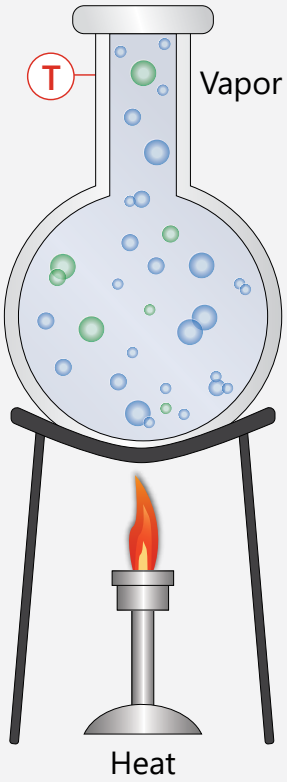
If the vapor pressure is ***too high***, it means that the product contains too much of lighter, lower boiling, material and vice versa.



Initial and End Boiling Point

Initial Boiling Point





End Boiling Point

Because no separation is perfect, there is always some of the other component in a distilled product stream.

For fractionation, a product always contains some of the next lighter and next heavier fraction.

Initial boiling point (IBP) is the temperature at which a product sample starts boiling. It indicates how much of the *lighter* component is in a product.
For example, a low initial boiling point in a bottoms product indicates too much of the overhead component in the product.

End boiling point (EBP) is the temperature at which a product sample is completely vaporized. It indicates how much of the *heavier* component is in a product.
For example, a high end boiling point in an overhead product indicates too much of the bottoms component in the product.

In fractionation, the IBP is equal to the cut point between the product and the next lighter fraction. The EBP is equal to the cut point between the product and the next heavier fraction.

Notes

Discuss with a Trainer

In a distillation system, the reboiler is responsible for _____ _____.

The ***temperature*** in a distillation column (increases/decreases) _____ top to bottom.

In a distillation system, the overhead condenser is responsible for _____.

The ***pressure*** in a distillation column (increases/decreases) _____ top to bottom.

The upper section of a distillation column is called the _____ section, the lower section is called the _____ section, and the feed entry section is called the _____ zone.

The concentration of the lower boiling point material (increases/decreases) _____ top to bottom in a distillation column.

Discuss with a Trainer

The material balance of a column is maintained when the _____ flow matches the _____ flow.

The role of trays or packings in a distillation column is to _____.

Discuss with a Trainer - Answer Key

The temperature at which a pure substance vaporizes is called the **boiling point**.

The bubble point of a mixture is the temperature at which **the first vapor bubble forms in a mixture**.

Heat that causes a phase change of a substance is called **latent heat**.

Batch distillation means **that all of the mixture is placed in a vessel at once and is exposed to heat at the same time.**

The boiling point of substance increases when the pressure **increases**.

Continuous distillation means **that feed is continuously supplied to the heated vessel and both products are continuously removed**

Discuss with a Trainer

Vapor-liquid equilibrium in distillation means that the amount of condensation is **same as** the amount of vaporization.

Reflux is important in distillation columns because **it provides cooling to a distillation column and is important for temperature control of the column.**

Discuss with a Trainer - Answer Key

In a distillation system, the reboiler is responsible **for providing most of the heat required for distillation**.

The ***temperature*** in a distillation column (increases/decreases) **increases** top to bottom.

In a distillation system, the overhead condenser is responsible for **condensing the vapor that exits the top of a column**.

The ***pressure*** in a distillation column (increases/decreases) **increases** top to bottom.

The upper section of a distillation column is called the **rectification** section, the lower section is called the **stripping** section, and the feed entry section is called the **flash** zone.

The concentration of the lower boiling point material (increases/decreases) **decreases** top to bottom in a distillation column.

Discuss with a Trainer

The material balance of a column is maintained when the **feed stream** flow matches the **overhead and bottom products** flow.

The role of trays or packings in a distillation column is to **intensify vapor-liquid contact**.