UNIT EIGHT
SEPARATING BY HEATING

Special Terms

Distillation: The separation of two or more liquids, with different boiling points, by evaporating them and condensing the resulting vapors.

Binary Distillation: The distillation of a mixture of two liquids.

Still: A piece of equipment used for distillation.

Distillation Column: A modern variety of a still that can be used for distillation of liquids as a continuous process.

Reboiler: A heat exchanger that uses either steam or hot furnace gases to revaporize material reaching the bottom of a distillation column.

Distillation Tray (Plate): A part of a distillation column through which rising vapor is intimately mixed with descending liquid.

Bubblecap Tray: A type of distillation tray.

Overhead Product (Overheads): The part of the condensed vapor, from the top of a distillation column, that is withdrawn from the process.

Bottom Product (Bottoms): The material withdrawn from the bottom of a distillation column.

Multicomponent Distillation: The distillation of a mixture of more than two substances. Distillation of petroleum is a common example of this process.

Reflux: The condensed vapor, from the top of a distillation column, that is returned to trickle down the column.

Reflux Ratio: The ratio between the amount of condensed vapor returned to the column as reflux and the amount withdrawn as overhead product.

Evaporator: A device for concentrating solutions by removing part of the liquid as a vapor. The process is called evaporation.

Multiple-effect Evaporator: A type of evaporator in which vapor from one stage of the system is used to heat liquid in another stage.

Vocabulary Practice

1. What is *distillation*?
2. How many different materials are separated in *binary distillation*?
3. What is a *still*?
4. What is a *distillation column*?
5. What is a *reboiler*?
6. What part of a distillation column is a *distillation tray*?
7. What is a *bubblecap tray*?
8. What is *overhead product*? What else is it called?
9. What is *bottom product*? What is it called?
10. Define *multicomponent distillation*. Give an example.
11. What is *reflux*?
12. What is the *reflux ratio* in a distillation column?
13. What is an *evaporator* used for?
14. What is a *multiple-effect evaporator*?

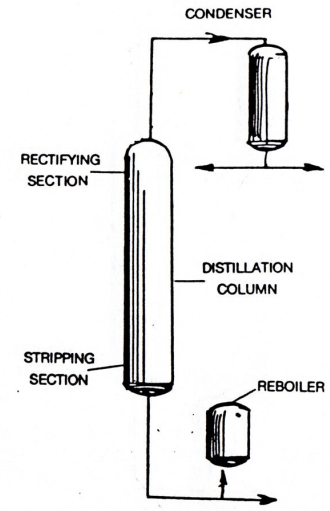
Separating by Heating

The chemical engineer often needs to separate mixtures of materials. These mixtures sometimes occur naturally; petroleum, for example, is a mixture of a great many chemicals. Some mixtures occur as a result of chemical reactions; few reactions will produce a single pure chemical. Often the desired material is produced with some that are undesirable; the resulting mixture must be separated so as to recover the wanted one in as pure a state as possible.

Distillation has been used to separate mixtures of liquids since the earliest days of chemistry. It is based on the principle that if a mixture of liquids is heated, some of the ingredients will evaporate faster than others and this property can be used to effect a separation. Let us imagine that we have a mixture of two liquids, A and B, in equal parts – the mixture contains 50% of Liquid A and 50% of Liquid B. If we heat the mixture until it boils, we may find that the vapor contains 75% of Liquid A and only 25% of Liquid B. Liquid A is evaporating faster than Liquid B.

In distillation, a liquid mixture is heated until it vaporizes, and the vapor is then condensed back into a liquid. Two liquids are generally quite easy to separate by distillation if the pure materials have boiling points that differ by a considerable amount, but modern techniques permit the separation of liquids whose boiling points are close together. Separation of mixtures of two liquids is called ***binary distillation***.

Distillation can be carried out as a batch process; this was the original kind of distillation and is still sometimes done today. For example, alcoholic beverages, such as brandy and some whiskies, are distilled by batch processes. (Essentially, these distillations consist of separating ethyl alcohol from water; consequently, they are binary distillations.) However, continuous distillation is faster and more efficient than the batch process, and most modern ***stills*** are of the continuous type. Continuous stills are called ***distillation columns*** because they look like circular architectural columns or large diameter pipes standing vertically on one end. But the interior of a pipe is empty space, and a distillation column is anything but empty.



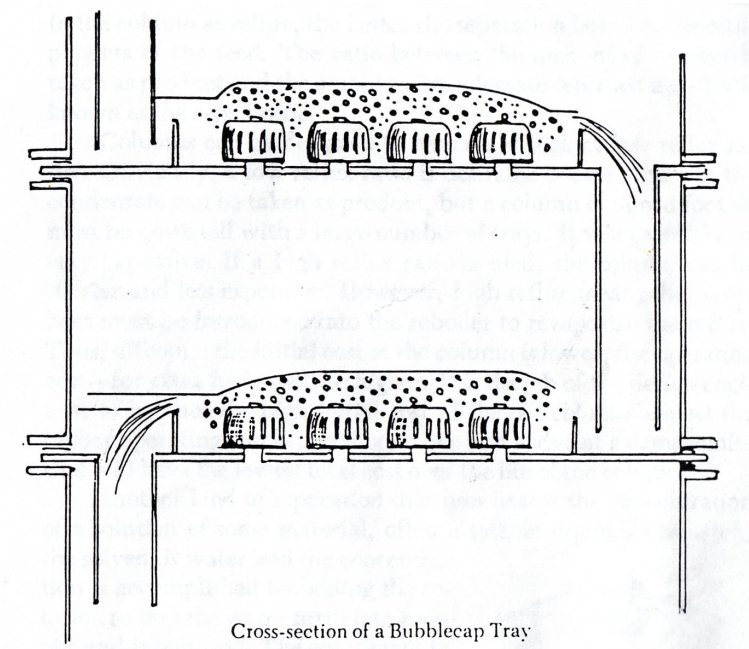
Distillation Column

Heat is supplied to a distillation column by a heat exchanger called a ***reboiler.*** Here liquid is vaporized and starts to ascend the column. At the top of the column, vapor is condensed in another heat exchanger (a condenser) and reintroduced into the column, where it starts to trickle downwards. Spaced at intervals throughout the column are flat ***plates***, usually called ***trays***, that are designed to ensure thorough mixing between the vapor that is ascending the column and the liquid that is descending. It is particularly easy to understand the process taking place on a ***bubblecap tray*** (although there are many that simpler to build and more efficient).

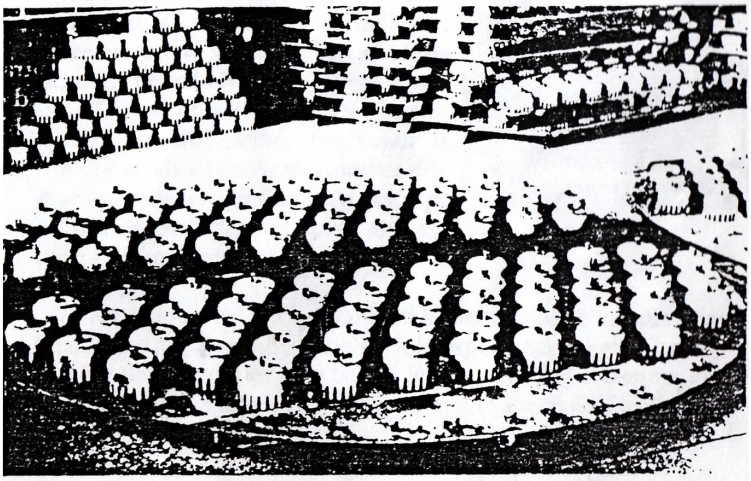
As shown in the diagram, the tray is covered with a layer of liquid. The lower edges of the bubble caps are below the surface of the liquid. Vapor rising in the column comes up under the bubble cap and is forced to bubble through the pool of liquid on the tray, thereby thoroughly mixing the vapor and the liquid. The liquid on the tray is cooler than the rising vapor; as the vapor bubbles through the liquid, any material in the vapor that is close to its condensation point (a temperature identical to its boiling point) will tend to condense and remain in the pool of liquid. At the same time the vapor, in cooling, is heating the liquid. Any component in the liquid that is near its boiling point tends to vaporize and be carried up the column.

A temperature gradient occurs along the height of the column, with the highest temperature at the bottom near the reboiler and the lowest at the top of the column near the condenser. After the column has been in operation for a time, the components of the mixture being distilled tend to distribute themselves on the various trays of

the column according to their boiling points – the materials with the lowest boiling points will be near the top.

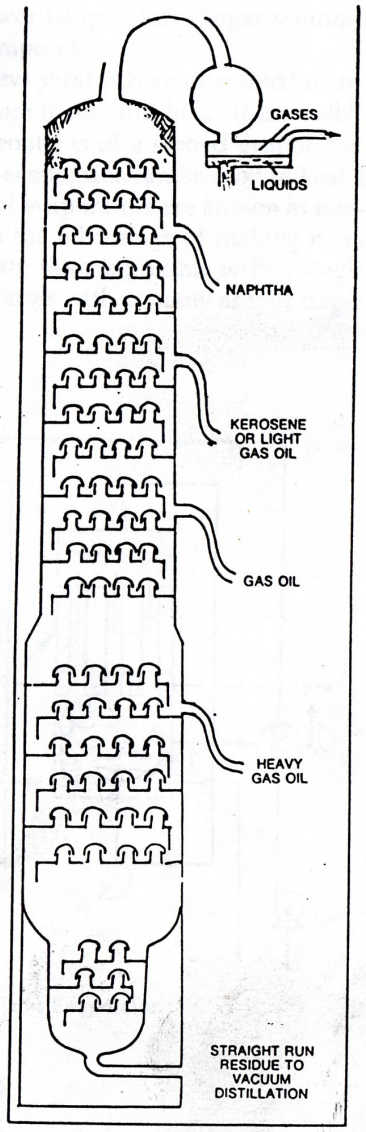


Cross-section of a Bubblecap Tray



Bubblecap Tray

Once the column is working, feed can be continuously introduced (usually near the middle) and product withdrawn from the top of the column, from the bottom, and at various intermediate points. If the material being distilled contains only two components (such as ethyl alcohol and water), all of the desired product (the alcohol) will be taken from the top of the column. This is called ***overhead product***, or simply ***overheads***. The water, which is unwanted, is taken from the bottom of the still and discarded. This is called ***bottom product***, or ***bottoms***. If the material being distilled contains many components (as is the case with petroleum), products of desired composition may be withdrawn from intermediate trays, as well as from the top and bottom of the still. This is known as ***multicomponent distillation***. The product withdrawn from intermediate trays may not be pure; it may be a mixture of several liquids with boiling points close together. Note that in petroleum distillation the bottoms are not thrown away (as was the case in the water/alcohol distillation) but are themselves valuable products. When the product withdrawn from one of the plates of a still is a mixture, it may be salable as such – kerosene,



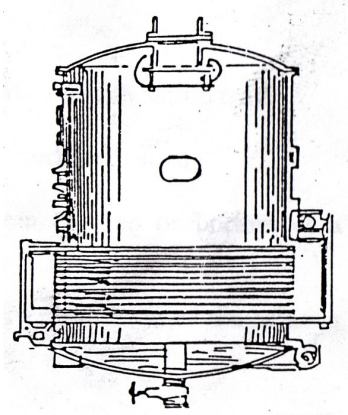
Multicomponent Distillation

gasoline, and benzine are such mixtures. However, if desired, the mixtures may be further separated in another still.

The liquid returned to the distillation from the overhead condenser is known as ***reflux***. In general, the more overheads returned to the column as reflux, the better the separation between the components of the feed. The ratio between the amount of overheads taken as product and the amount of condensate returned as reflux is known as the ***reflux ratio.***

Columns can be designed to operate at high or low reflux ratio. Obviously, a low reflux ratio is desirable because more of the condensate can be taken as product, but a column designed for this must be quite tall with a large number of trays. It will therefore be very expensive. If a high reflux ratio is used, the column can be shorter and less expensive. However, high reflux means that more heat must be introduced into the reboiler to revaporize the reflux. Thus, although the initial cost of the column is lower, the operating cost – for extra fuel – will be higher. It is the job of the design engineer to balance the high initial cost of the tall column against the higher operating cost of the shorter one and arrive at a compromise that will have the lowest total cost over the life of the column.

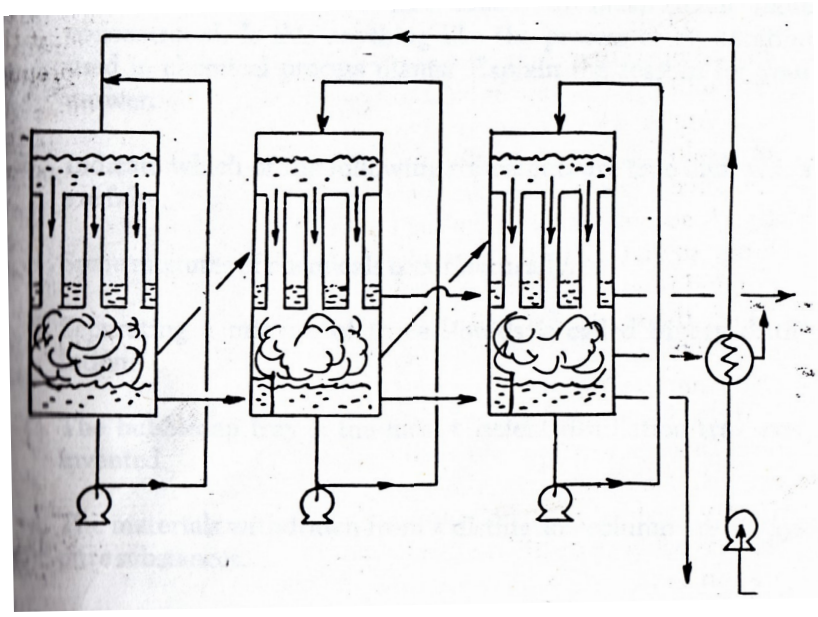
Another kind of separation that uses heat is the concentration of a solution of some material, often a salt, in liquid. Most often, the solvent is water and the concentration is accomplished by boiling the solution so that the water turns into a vapor and is removed. The equipment in which the solution is boiled and concentrated is known as an ***evaporator***. In ***evaporation***, what remains after the liquid is boiled off is the desired product. The vapor has no value and is discarded. In distillation, the vapor is usually a valued product. Another difference between distillation and evaporation is that there is never any attempt to separate the components of the vapor.



Evaporator

An evaporator is a special kind of heat exchanger, usually vertical. Heating is usually done with low-pressure steam. The vapor leaving the evaporator may be condensed, creating a vacuum in the evaporator. The effect of the vacuum is to make the liquid in the evaporator boil faster and at a lower temperature. Sugar solutions are usually concentrated in such evaporators.

Although the vapor from an evaporator is an unwanted material, it does have some value because it contains heat. It is possible to use the hot vapor to heat the contents of a second evaporator. Sometimes vapor from the second evaporator can be used to heat a third one, and so on. These series of evaporators are known as ***multiple-effect evaporators***, and have the advantage of making more efficient use of the heat in the steam than is possible with a single evaporator. Multiple-effect evaporators with as many as four stages are common.



Three-effect Vertical Tube Evaporator

Discussion

1. Why do chemical engineers need to separate mixtures of liquids?
2. What is the principle on which distillation is based?
3. Is distillation a batch or continuous process?
4. Give an example of binary distillation.
5. Are distillation columns continuous stills?
6. How is heat supplied to a distillation column?
7. What is used to condense vapors from a distillation column?
8. What is the purpose of a distillation column tray?
9. Name one kind of distillation tray. Describe it.
10. Describe that process that goes on at each distillation tray.
11. From what part of the column are overheads taken?
12. Are products withdrawn only from the top or bottom of a column?
13. Are the products taken from intermediate trays of the column always single pure materials?
14. Are bottoms always waste products?
15. Where does reflux come from?
16. Is a high reflux ratio preferable to a low one? Why?
17. What is a disadvantage of designing a column to use a low reflux ratio?

18. What are some differences between distillation and evaporation?

19. How are evaporators usually heated?

20. Is there any way to use the vapor from an evaporator?

21. What is the disadvantage of a multiple-effect evaporator?

Review

A. Cooks often boil soups and sauces to make them more concentrated. Is this anything like the process of evaporation used in chemical process plants? Explain the reasons for your answer.

B. Indicate which of the following statements are <u>true</u> and which are <u>false</u>.

1. Some mixtures of chemicals occur naturally.
2. Separating a mixture of three liquids is called binary distillation.
3. The bubble-cap tray is the most efficient distillation tray ever invented.
4. The materials withdrawn from a distillation column are always pure substances.
5. A low reflux ratio makes a distillation column cheaper to operate.
6. In both distillation and evaporation, a liquid is changed into a vapor.
7. Multiple-effect evaporators are less efficient than single evaporators.

8. The vapor from a multiple-effect evaporator is called reflux.

9. Multicomponent distillation is used in processing petroleum.

10. Sugar solutions are often evaporated with the help of a vacuum.

11. The liquid to be distilled is always fed into a distillation column at the top.

12. Sometimes the product of one distillation column is redistilled in another column.

13. While a still is operating, a bubble-cap tray is covered with a layer of liquid.

14. Heat is supplied to a distillation through a heat exchanger called a condenser.

15. When vapor bubbles through the liquid on a distillation tray, some of the vapor is condensed and some of the liquid vaporizes.

16. Distillation can be carried out either as a continuous process or a batch process.