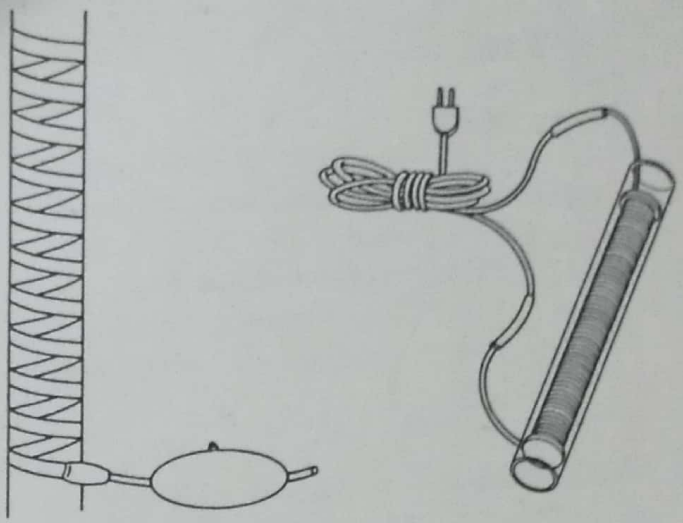
Heating Fractionating Columns

Sometimes a fractionating column must be heated in order to achieve the most efficient fractionation of distillates. This may be accomplished by wrapping the column with heating tape or a resistance wire, such as Nichrome wire, controlled by variable transformers (see Fig. 21.13). (Refer to the section on heating mantles in Chap. 10, "Heating and Cooling.")



(a) (b)

FIGURE 21.13
Methods of heating fractionating columns. (*a*) Using heating tape. (*b*) Nichrome wire heater.

Efficiency of Fractionating Columns

We measure the efficiency of a fractionating column in terms of the number of theoretical plates that it contains. A column with one theoretical plate is one in which the initial distillate has a vapor composition that is at equilibrium with the original solution. It is impossible to operate fractionating columns at equilibrium.

Bubble-Cap Fractionating Columns

Bubble-cap fractionating columns (Fig. 21.14) have definite numbers of trays or plates and are fitted with either bubble caps or sieve perforations—or modifications of these two—to enable the achievement of intimate vapor-liquid dispersion. However, because fractionating columns cannot be operated at equilibrium (practically), the number of theoretical plates is always lower than the number of actual plates, depending upon the rate of distillation, reflux ratio, and other factors.

CAUTION If you heat the pot too vigorously and remove the condensed vapor too quickly, the whole column will heat up uniformly and there will be no fractionation. The fractionating column will become flooded by the returning condensate.

Total-Reflux–Partial-Takeoff Distilling Heads

Exercise good judgment in the control of the amount of heat applied, and, for truly effective fractionation, use a total-reflux–partial-takeoff distilling head as shown in Fig. 21.15. With the stopcock S completely closed, all condensed vapors are returned to the distilling column, a total reflux condition. With the stopcock par-

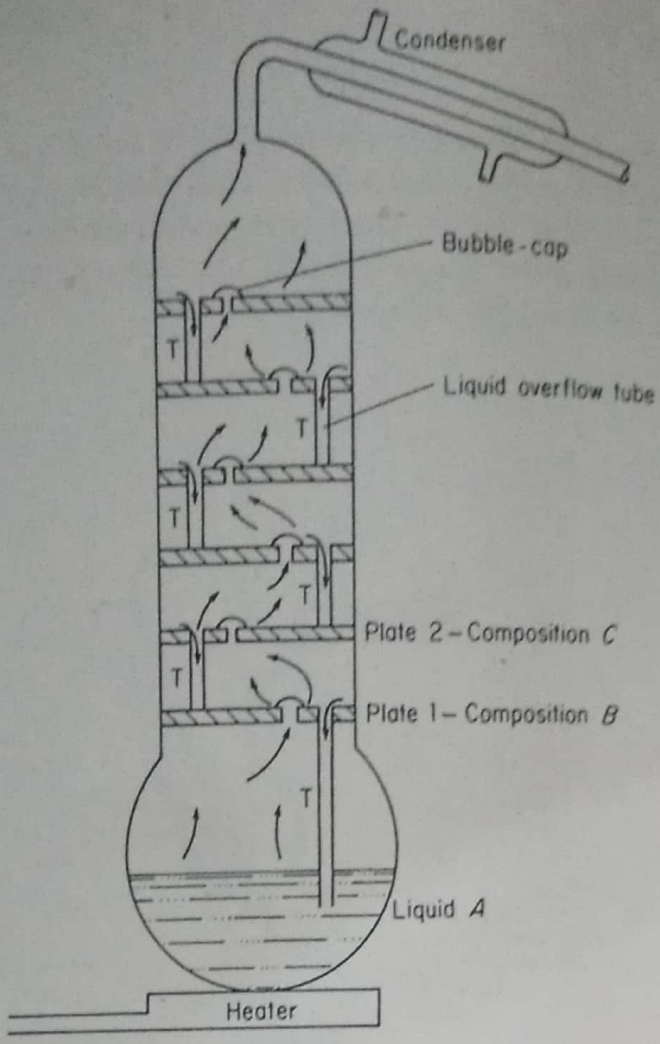


FIGURE 21.14
Bubble-cap fractionating column.

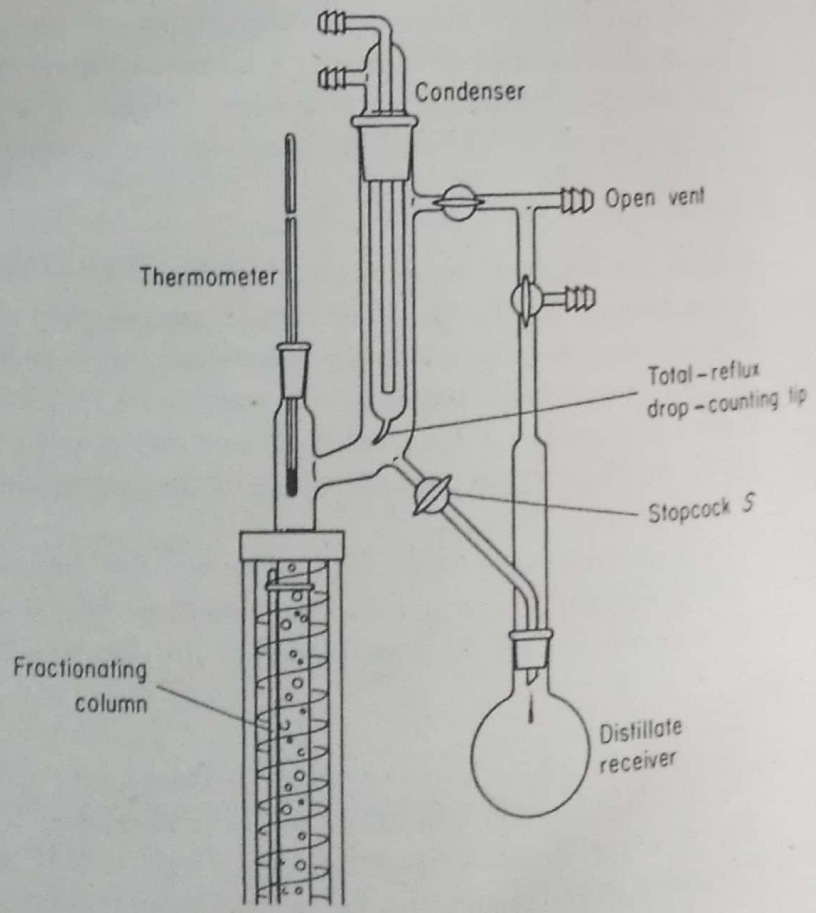


FIGURE 21.15
Total-reflux–partial-takeoff still head.

tially opened, the number of drops of condensate falling from the condenser which returns to the fractionating column can be adjusted. The ratio of the number of drops of distillate allowed to pass through stopcock S into the receiver to the number of drops of reflux is called the *reflux ratio*. With an efficient column, reflux ratios as high as 100 to 1 can be used to effectively separate compounds that have very close boiling points.

VACUUM DISTILLATION

Principle

Many substances cannot be distilled satisfactorily at atmospheric pressure because they are sensitive to heat and decompose before the boiling point is reached. Vacuum distillation, distillation under reduced pressure, makes it possible to distill at much lower temperatures. The boiling point of the material is affected by the pressure in the system. The lower the pressure, the lower the boiling point; the higher the pressure, the higher the boiling point.

Nomographs

Nomographs are special graphs that enable the technician to determine more accurately the boiling points at different pressures; they also provide a method of converting boiling points to the pressure desired.

Procedure for using the nomograph (see Fig. 21.16):

1. Select the desired boiling point at the reduced pressure.

2. Use a transparent plastic ruler and connect that boiling point (column A) with the given corresponding pressure (column C). It will intersect column B at a definite point. Record that point.

3. Using the point obtained from column B in step 2, select the new pressure desired on column C. Align the plastic ruler with these two points and read the corresponding temperature for the boiling point at the new pressure where the ruler intersects column A.

EXAMPLE A liquid boils at 200°C at 10 torr pressure. What would be (1) the normal boiling point at 760 torr and (2) the boiling point at 1 torr pressure?

Solution Given: bp = 200°C (column A) at 10 torr (column C).

1. Intersection point in column B is 350°C, the boiling point at 760 torr pressure (corrected).

2. The line connecting the points 350°C on column B, and 1 torr pressure on column C intersects column A at 150°C. Thus 150°C is the boiling point at 1 torr pressure.

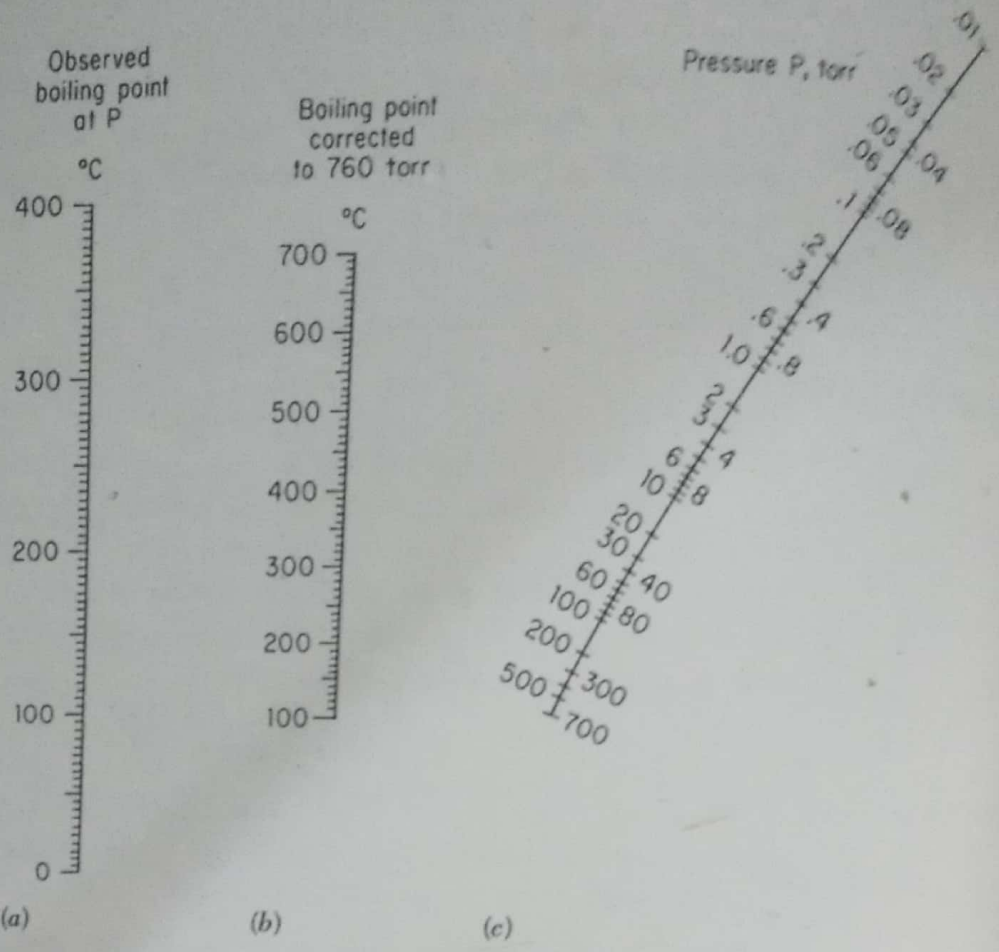


FIGURE 21.16
Nomograph for pressure and temperature at various boiling points.

CAUTION Glass equipment may collapse under reduced pressure. Use *safety glasses* and a *safety shield* as well as special equipment shown in Fig. 21.17.

General Requirements

The experimental setup is as shown in Fig. 21.18.

1. *A source of vacuum.* Efficient water pumps, aspirators, will theoretically reduce the pressure in the system to the vapor pressure of the water passing through the pump. In practice, the pressure is usually about 10 mm higher. (*a*) Oil mechanical vacuum pumps. (*b*) Use rubber pressure tubing. (*c*) The entire distillation system should be airtight, free from leaks. (*d*) Lubricate all joints and connections.

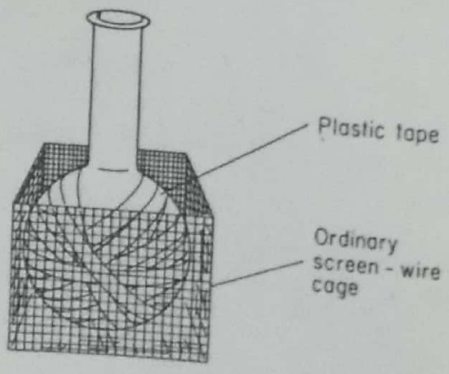


FIGURE 21.17
Safety precautions for a large flask in a vacuum system.

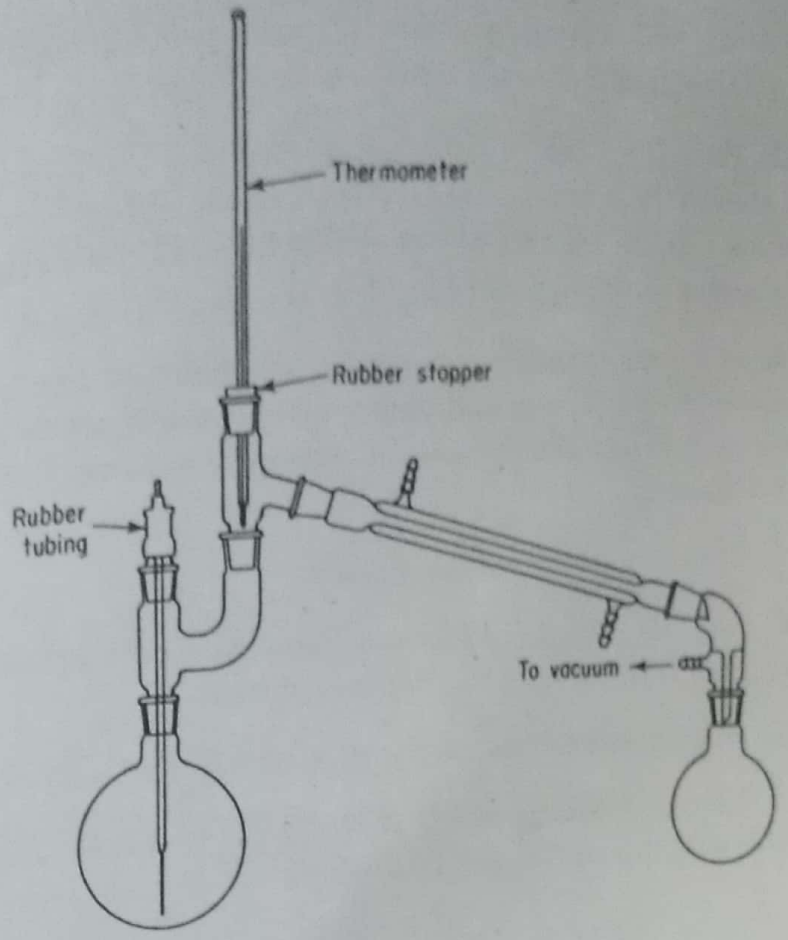


FIGURE 21.18
Vacuum distillation with gas-capillary bubbler.

2. *Safety trap* to protect manometer and vacuum source from overflow-liquid contamination (Fig. 21.19). The trap must be correctly connected. Vapors condense on the sides of the trap and fall to the bottom if they are not solidified.

Dry ice will freeze your skin; *do not handle with bare fingers or hands.*

(**a**) Crush dry ice in a cloth towel with a hammer.

(**b**) Use a scoop to fill the Dewar flask after the trap has been inserted.

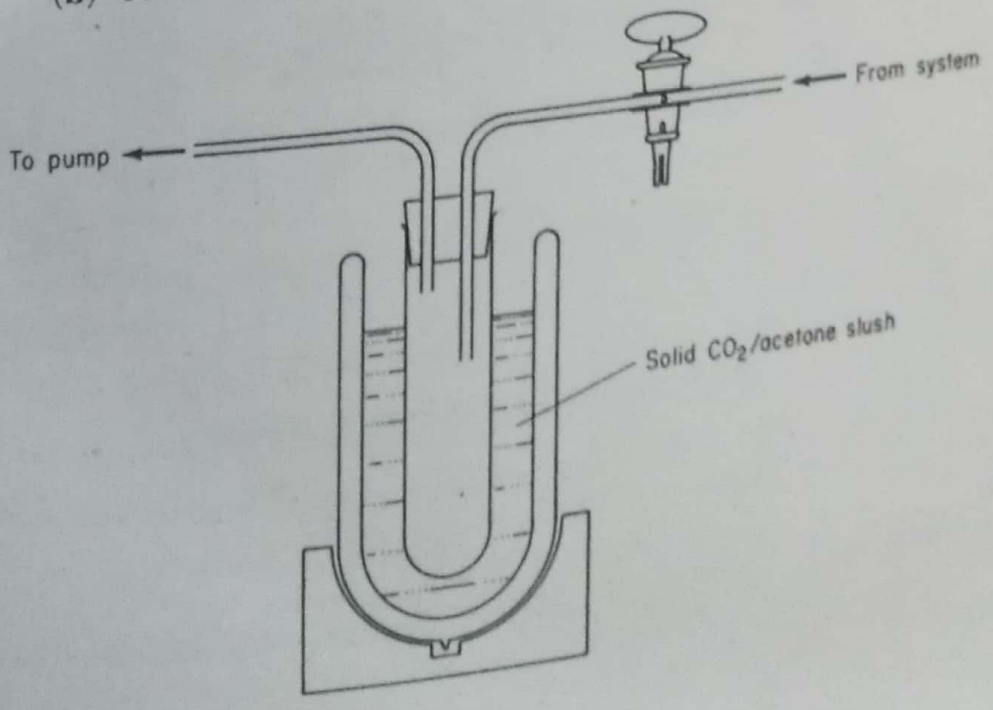


FIGURE 21.19
Dry-ice vapor trap.

(c) Add solvent, acetone, or isopropanol in small increments until the trap is filled and the liquid level is near the top.

3. *Pressure gauge* (manometer). Exercise great care when allowing air into the evacuated system. It must be done slowly to avoid breakage when the mercury column rises to the top of the closed tube. (Refer to the section on manometers in Chap. 5, "Pressure and Vacuum.")

4. *Manostat* (pressure regulator). To maintain constant pressure in the system, it automatically opens and closes needle valves, permitting air to enter or keeping the system airtight because of vacuum variations. Refer to Chap. 5, "Pressure and Vacuum."

5. *Capillary air inlet* (Fig. 21.20).

6. *Special vacuum distillation* flasks to minimize contamination of the distillate caused by frothing of the boiling solution.

7. *Heating baths, electric mantles,* and *fusible alloy* or *sand baths.*

8. *Special distilling heads* (Fig. 21.21) to permit removal of distillate fractions without interrupting the distillation.

FIGURE 21.20
Capillary gas- or air-inlet tube.

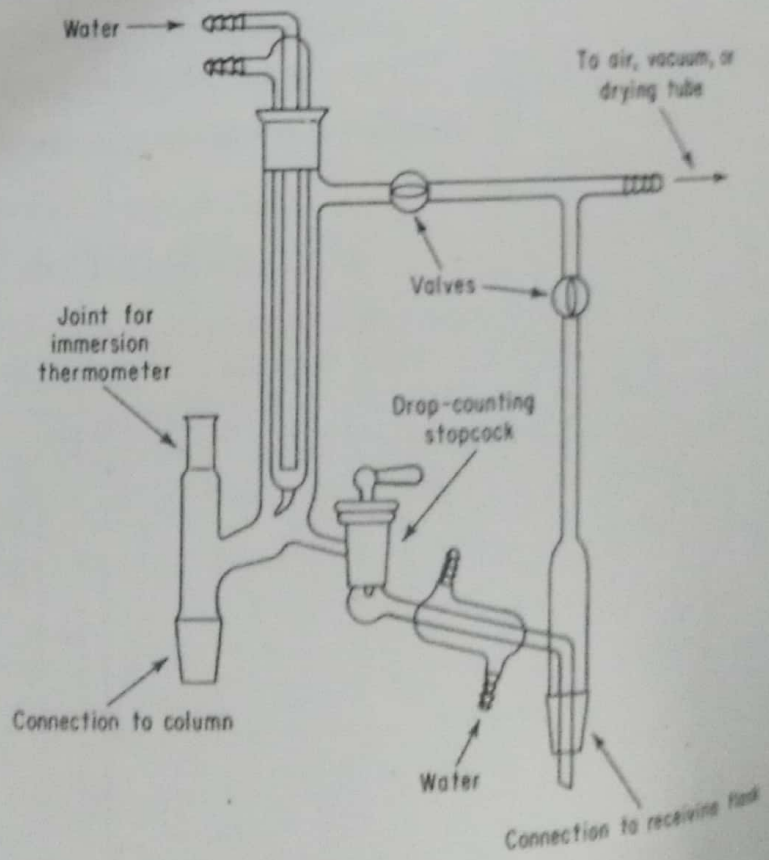


FIGURE 21.21
Partial-takeoff distillation head and its component parts.

Assemblies for Simple Vacuum Distillation and Fractionation

Simple Vacuum Distillation

Procedure

A *Claisen flask* for use in this process is shown in Fig. 21.22.

1. Fill the Claisen flask (*A*) one-third full.
2. Apply vacuum; adjust the capillary air inlet (*C*) with the pinch clamp (*D*).

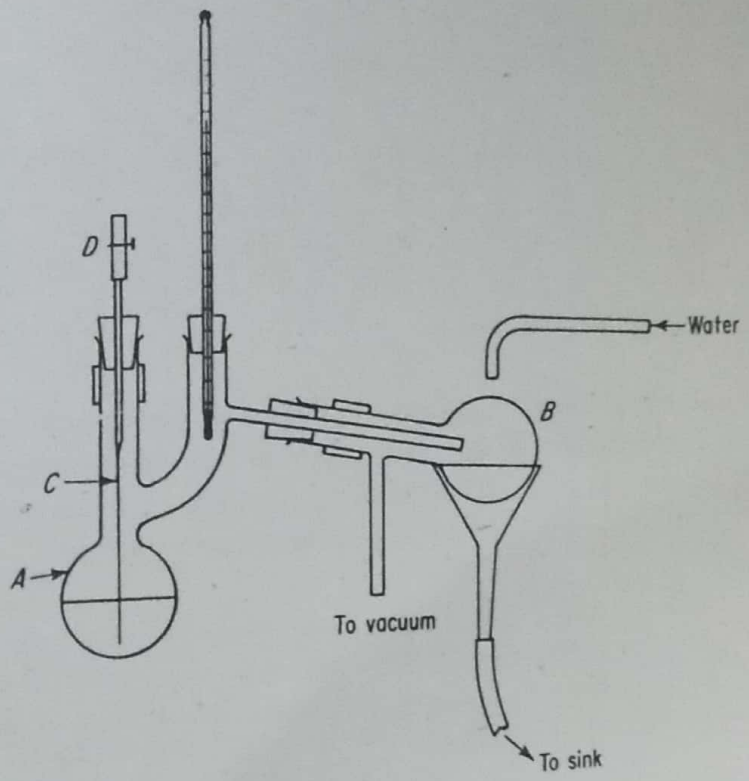


FIGURE 21.22
Claisen flask setup for vacuum distillation.

3. Heat bath to about 20°C higher than the temperature at which the material will distill.
4. Cooling water flowing over the receiver (*B*) condenses the vapors to give a distillate.
5. A safety trap prevents any condensate from contaminating the suction pump or manometer.
6. When distillation is completed:
   (a) Remove the heating bath; allow the flask to cool.
   (b) Remove the capillary pinch clamp.
   (c) Cut off the cooling water.
   (d) Turn off the vacuum pump.

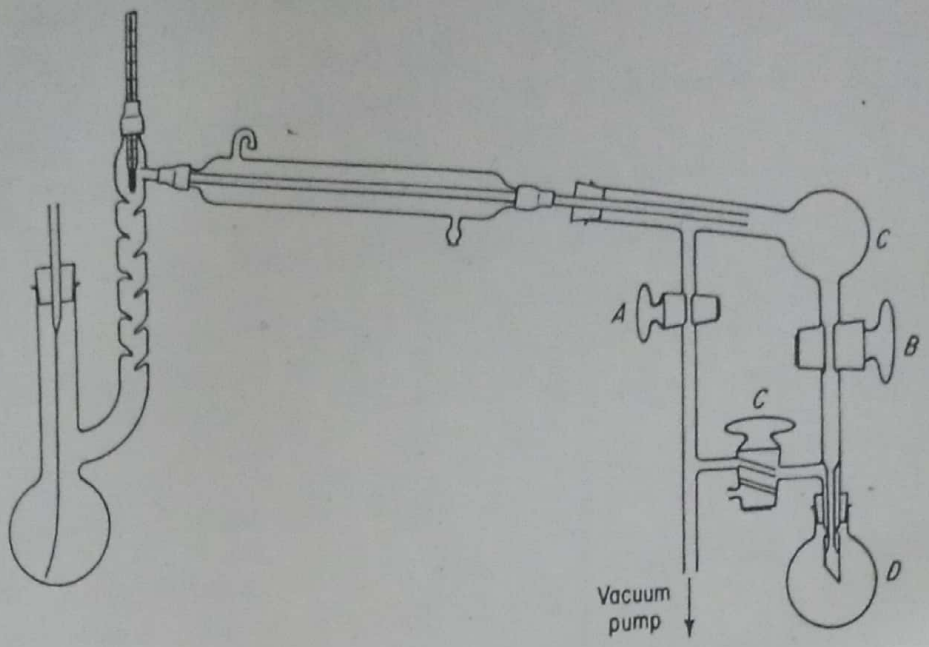


FIGURE 21.23
Claisen apparatus for fractional distillation in a vacuum.

Vacuum Fractionation

A *Claisen fractionating column* is shown in the apparatus in Fig. 21.23. The distillation neck of the Claisen flask serves as a fractionating column because of the indentations. The receiver makes it possible to remove distillate fractions without interrupting the distillation or breaking the vacuum.

Procedure

Follow the steps and observe the cautions previously listed. To remove distillate fractions during distillation:

1. Close stopcock B.
2. Close stopcock C.
3. Reverse rotation stopcock C so that air flows into flask D.
4. Gently remove flask D, empty into bottle, and replace.
5. Rotate stopcock C 180° so that D is now under vacuum.
6. Open stopcock B to allow collected distillate to drain into D.

Complete the fractional distillation and disconnect the equipment as described above.

Modified Distillate Receiver

Collecting individual distillate fractions without removing the receiver (as in the procedure above) can be accomplished with the modified receiver. The receiver is adjusted to collect the particular fraction desired and then rotated to meet the need. This method does not introduce any possible problems that might occur in

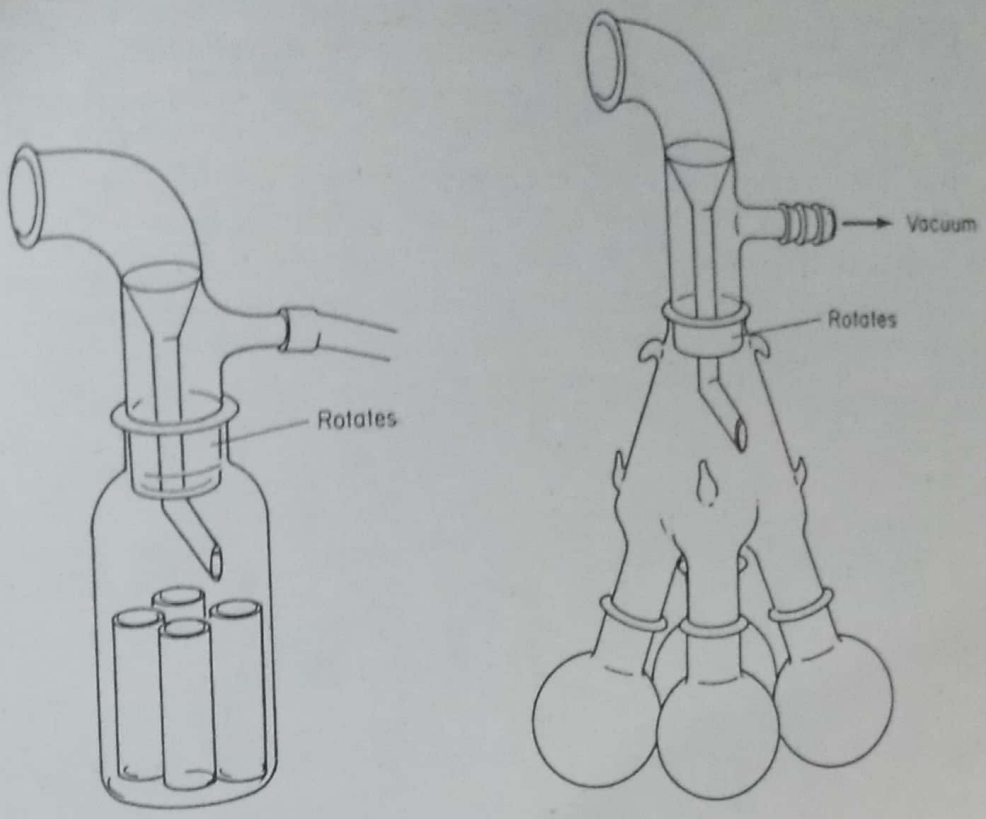


FIGURE 21.24
Modified receivers for collection of distillate fractions.

the procedure if the vacuum distilling pressure is affected when the receiver is removed to isolate the distillate fraction. (See Fig. 21.24.)

A more complex apparatus is shown in Fig. 21.25.

STEAM DISTILLATION

Principle

Steam distillation is a means of separating and purifying organic compounds by volatilization. The organic compound must be insoluble or slightly soluble in water. When steam is passed into a mixture of the compound and water, the compound will distill with the steam. In the distillate, this distilled compound separates from the condensed water because it is insoluble in water.

Most compounds, regardless of their normal boiling point, will distill by steam distillation at a temperature below that of pure boiling water. For example, naphthalene is a solid with a boiling point of 218°C. It will distill with steam and boiling water at a temperature less than 100°C.

Some high-boiling compounds decompose at their boiling point. Such substances can be successfully distilled at low temperature by steam distillation.

Steam distillation can be used to rid substances of contaminants because some water-insoluble substances are steam-volatile and others are not.

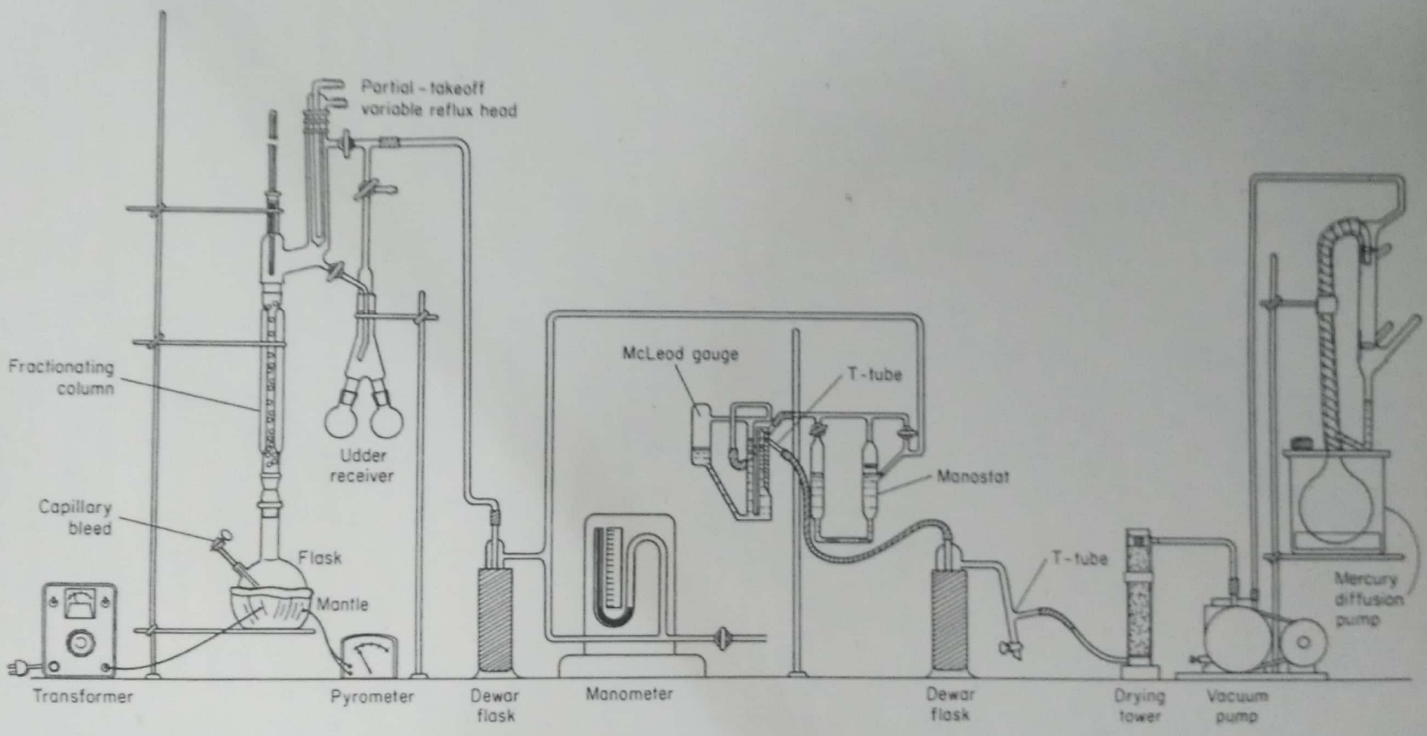


FIGURE 21.25
Apparatus setup using a diffusion pump for high-vacuum fractional distillation.

When it is desirable to separate nonvolatile solids from high-boiling organic solvents, steam distillation will remove all solvents (water-insoluble).

Procedure

1. Place the compound or mixture in the distilling flask with a little water. Pass cooling water through the condenser (see Fig. 21.26). A Claisen flask may be substituted for the round-bottomed flask. The Claisen still head helps to prevent any contamination of the distillate caused by spattering of the steam-distilled mixture. If there is no readily available source of piped steam, the steam can be generated in an external steam generator (Fig. 21.27) and then passed into the mixture to be steam-distilled.

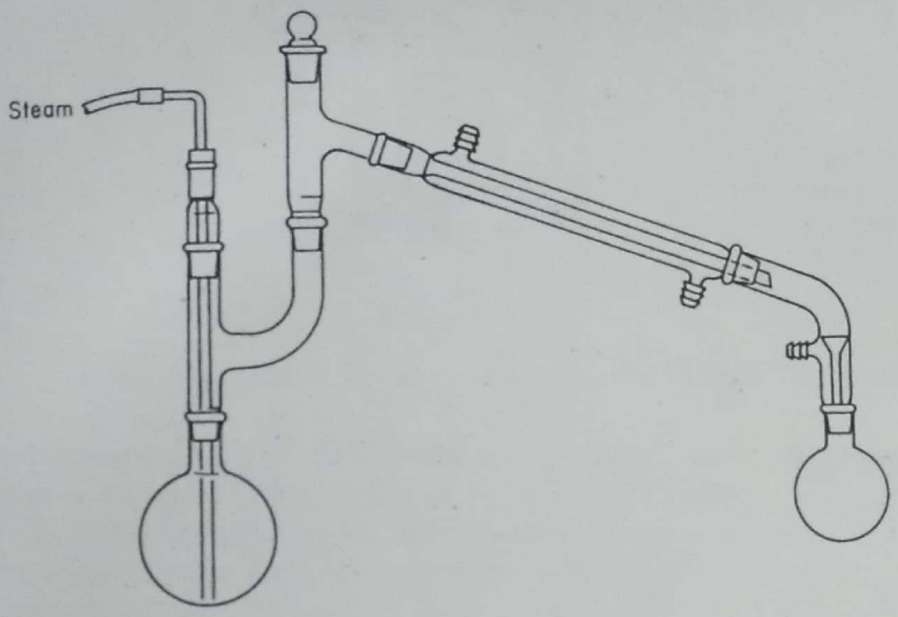


FIGURE 21.26
Apparatus for steam distillation.

CAUTION Always equip the steam generator with a safety tube to prevent explosions.

NOTE If only a small amount of steam is needed, for instance, to rid a substance of a small amount of steam-volatile impurity, water can be combined with the material directly in the distilling flask; the flask is then directly heated with a Bunsen flame or any other suitable heat source. (Refer to Chap. 10, "Heating and Cooling.") The long steam-inlet tube is replaced with a stopper.

2. Pass steam into the distilling flask with the steam outlet below the surface of the liquid. The distilling flask *itself* may be heated gently with a burner. If steam is available from a laboratory steam line, insert a water trap (Fig. 21.28) in the entering steam line to trap condensed water. Otherwise, the condensed water may fill up the distilling flask.

3. Continue passing steam into the flask until no appreciable amount of water-insoluble material appears in the condensate.