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# **DISTILLATION PROCESSES**

## **DISTILLATION PROCESSES**

Distillation is the process of converting liquid into its vapours by heating and reconvertng it again into liquid by condensing the vapours. It is a method of separating substances which differ in their vapour pressures.

The distillation process is carried out in an apparatus which consists of

- (a) Still, in which volatile material is boiled.
- (b) Condenser, in which vapours are condensed.
- (c) Receiver, in which distillate is collected.

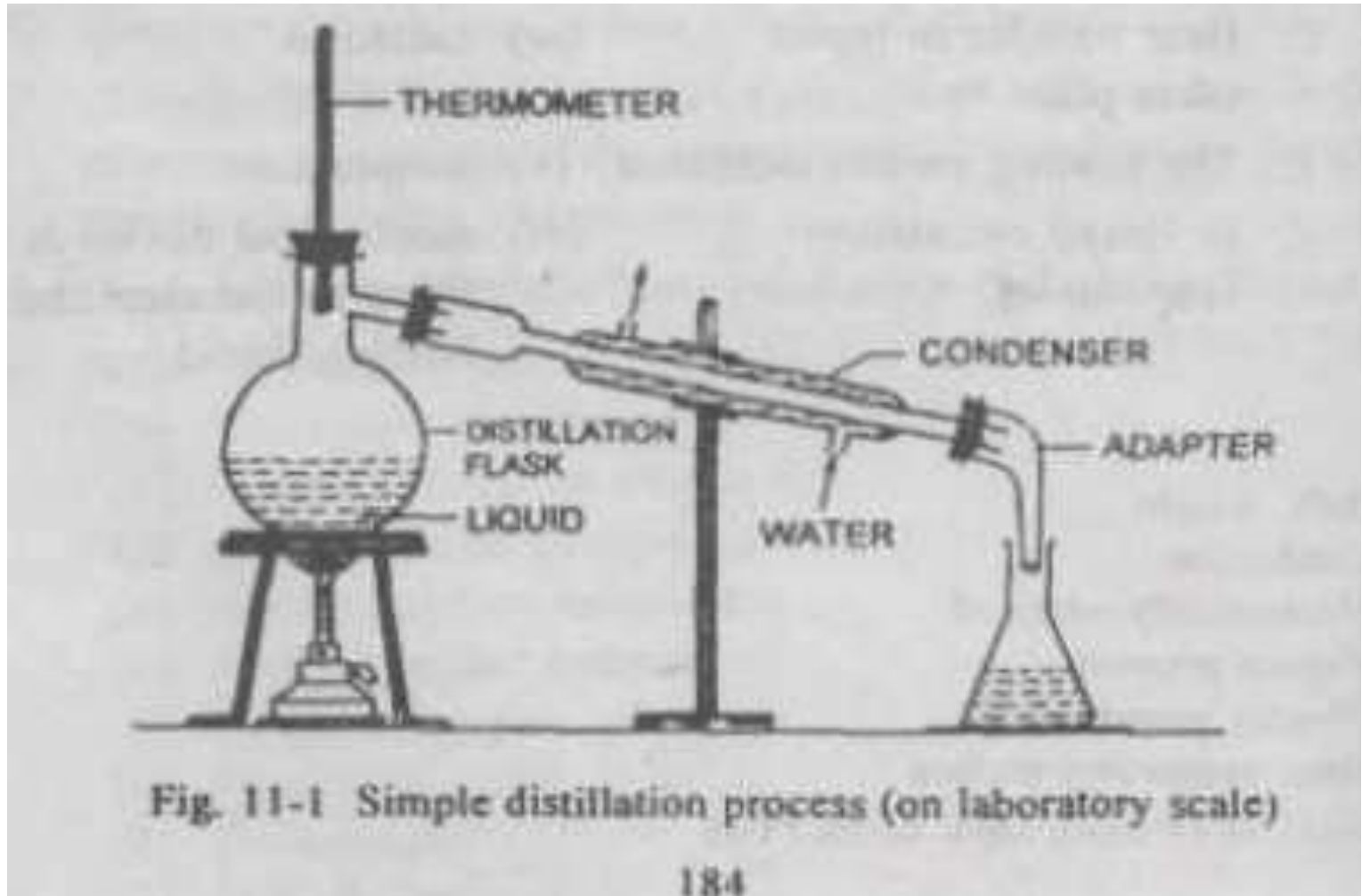
### **TYPES OF DISTILLATION PROCESSES**

The following are the various types of distillations:

1. Simple distillation
2. Distillation under reduced pressure
3. Fractional distillation
4. Steam distillation
- 5 . Destructive distillation

# 1. Simple Distillation

It is a process of converting a liquid into its vapour in a distillation still, transferring the vapour to another place and condensing it again into liquid.



Apparatus used for laboratory scale It consists of a distillation flask with a side arm sloping downward which is connected to a condenser.

The condensed vapours are collected in a flask called 'receiver'.

The whole apparatus is made of glass (see *Fig. 11-1*).

The distillation flask should be of such a size that it can contain half to two-thirds of the liquid to be distilled. The thermometer is fitted in distillation flask to note down the temperature, at which the vapours are distilled. Bumping is avoided by adding small pieces of porcelain or porous pot before distillation.

### **Applications of simple distillation in pharmacy**

1. It is used for the preparation of distilled water and water for injection.

2. Many volatile oils and aromatic waters are prepared by simple distillation.

3. Organic solvents are purified by distillation.

4. Many official compounds are prepared by distillation e.g.

*Spirit*

## 2. Distillation Under Reduced Pressure

Theory Liquid boils when its vapour pressure is equal to the atmospheric pressure. The boiling point of the liquid may be lowered to the desired temperature by reducing the pressure on its surface.

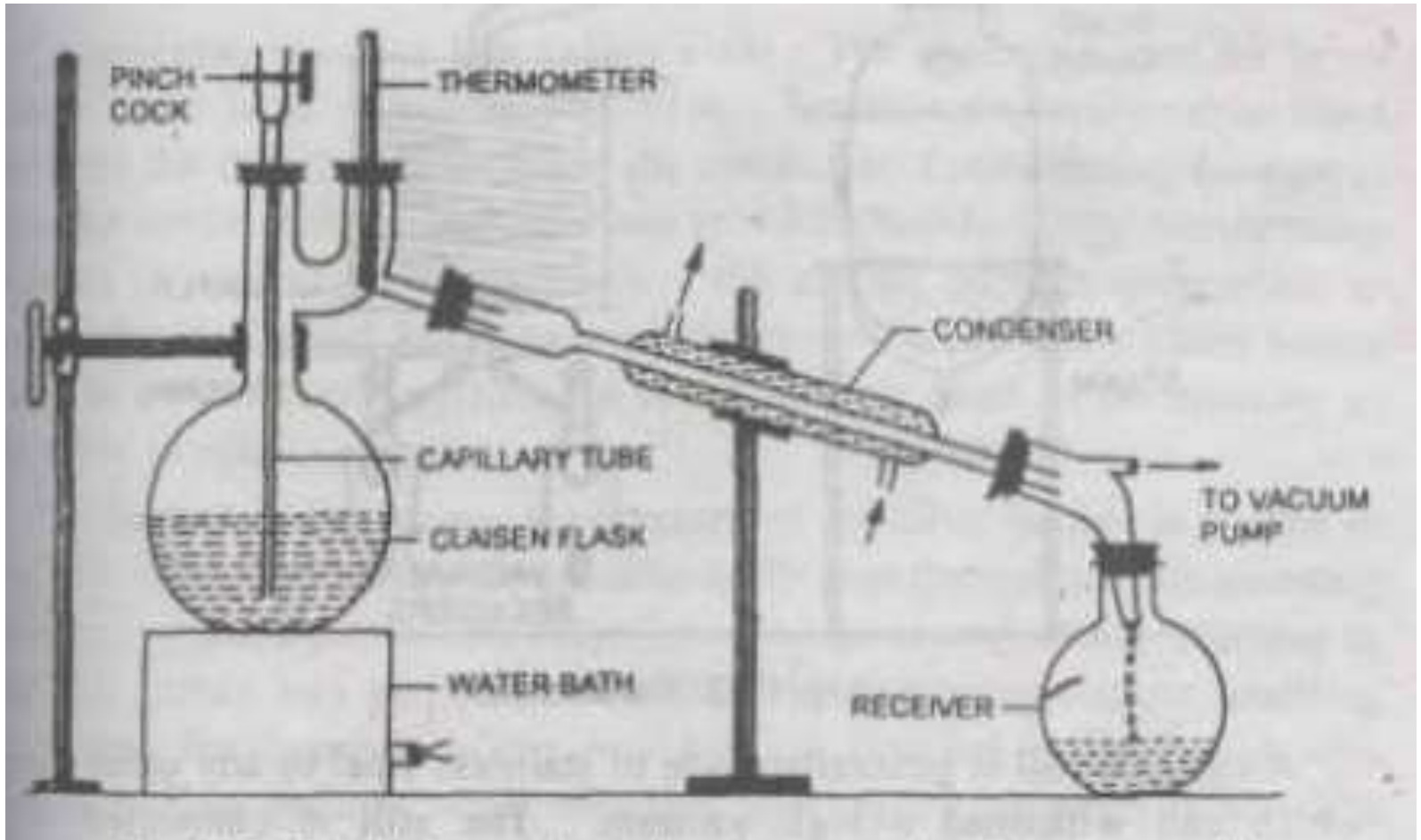


Fig. 11-2 Distillation under reduced pressure (on laboratory scale)

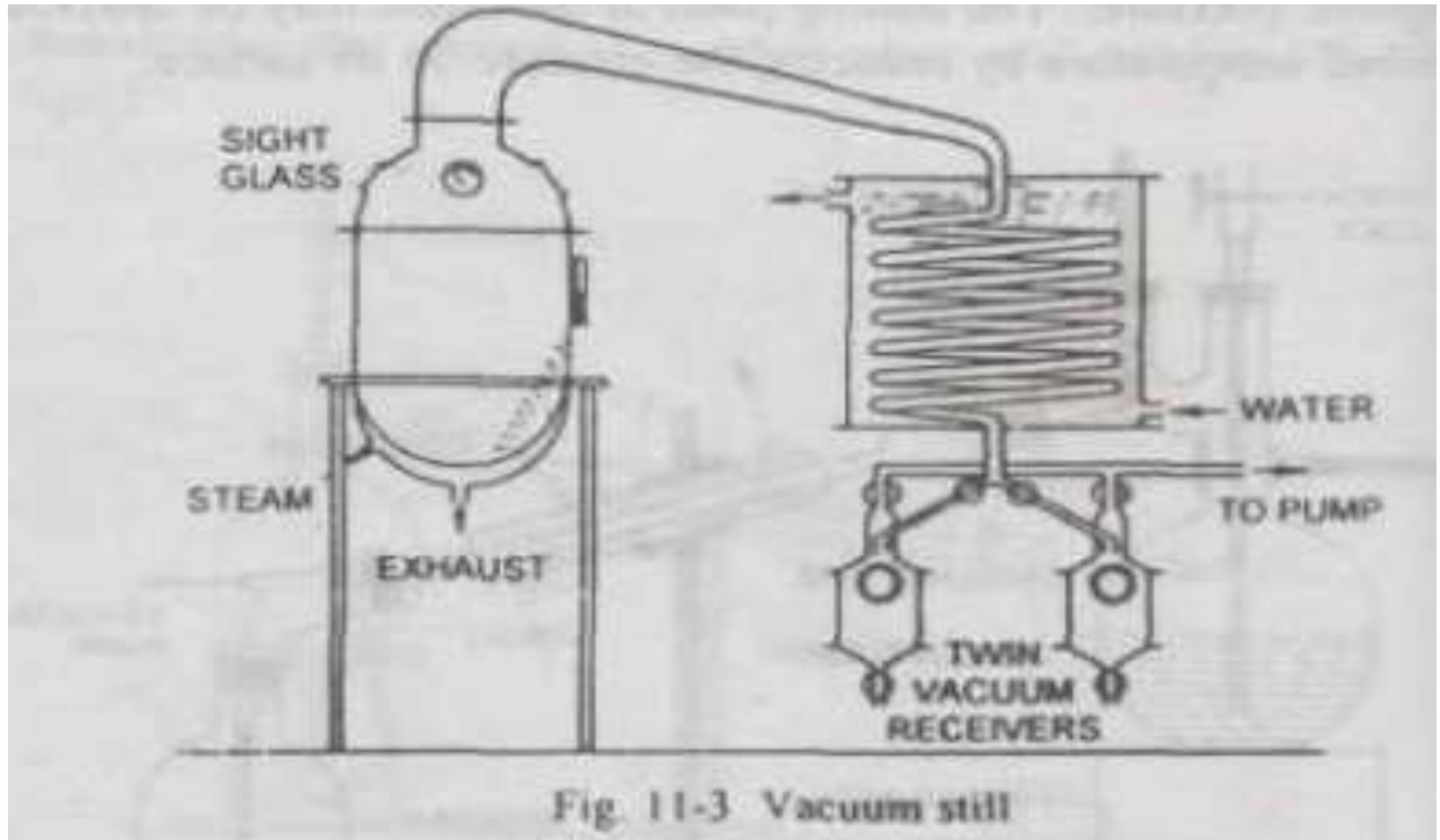
Apparatus used for laboratory scale It consists of a double-neck distillation flask known as "claisen flask'. In one of its necks a these mometer is fitted and in the second neck a capillary tube is fitted which prevents bumping of the heated liquid. The capillary tube should be so line as to permit only a slow stream of bubble which can be controlled with a pinch cock. Thick walled glass apparatus with inter-changeable standard glass joints are used for vacuum distillation. The claisen flask is connected to a receiver through condenser. Vacuum pump is attached to the receiver to attain the desired degree of vacuum. Heating of claisen flask is not started-until the desired vacuum has been attained.

### **Applications in pharmacy**

- 1.It is used for the concentration of extracts containing thermolabile (constituents which are sensitive to heat) m order to prevent their destruction.
- 2.It is used for separating substances which undergo decomposition when heated under normal atmospheric pressure.
3. It is used for obtaining a light porous mass on distillation of

## VACUUM STILL

The vacuum stills are employed for distilling substances under reduced pressure on a large scale.



A vacuum still is generally made of stainless steel or any other metal which can withstand a high vacuum. The still is connected to condenser. The vacuum is created by means of a vacuum pump.

- vacuum still is filled by attaching a pipe to a tap in the lower part of the hood and the pump is started.
- The other end of the pipe dips into the liquid to be distilled so that it can be drawn into the still.
- An observation window in the hood is very helpful to the operator to see the progress of distillation and also the level of the content of the liquid to be distilled.
- Two receivers are generally attached to the condenser in order to collect the distillate without stopping distillation. However, they may be used alternately by a suitable arrangement of the cocks.

### **Applications in pharmacy**

1. Distillation of substances that have a high boiling point at atmospheric pressure.
2. Distillation of thermolabile substances that get damaged by a high temperature.
3. Removal of the last traces of a volatile solvent.



### **3. Fractional Distillation (Distillation of miscible liquids)**

**Theory** When a substance is dissolved in a liquid, the vapour pressure of the liquid is lowered. When two miscible liquids are mixed together, each will act as solute or solvent for the other. So, when a mixture of two such liquids is heated, the vapour pressure of each is lowered. The pressure exerted by each liquid in the mixture is known as "partial pressure". "The liquid boils when the sum of the partial pressures is equal to the atmospheric pressure. The vapours arising from two miscible liquids at boiling point is richer in the component exerting the greater partial pressure.

**Apparatus used for laboratory scale** The apparatus used for laboratory scale is as shown in Fig. 11-4. Fractionating column is fitted between the distillation flask and the condenser. Fractionating column is used for continuous separation of two miscible liquids. Long fractionating column is used in the mixture where the boiling point is quite close to each other and short fractionating column is used in those cases where there is a considerable difference in the boiling point of the mixture of miscible liquids

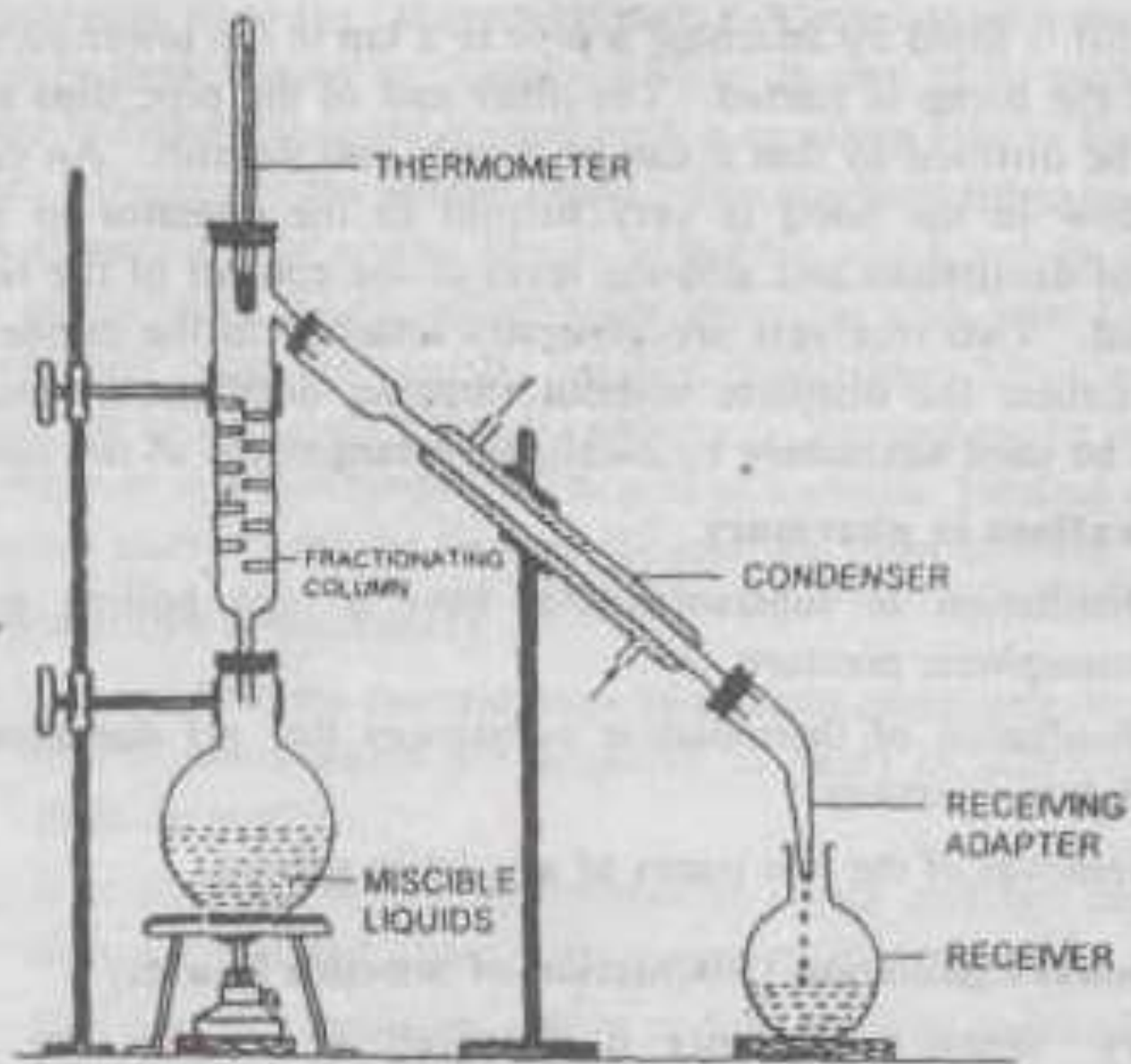


Fig.11-4 Fractional distillation (on laboratory scale)

In fractional distillation, the mixture of miscible liquids is heated in the still. The vapours formed are allowed to pass through the fractionating column, where a part of the vapour is condensed and while returning to the still comes into an intimate contact with the rising vapour resulting in further fractionation of the liquid being distilled. The liquid with higher boiling point is condensed first and vapour becomes richer with the liquid having the lower boiling point which gets condensed in a condenser.

#### Applications in pharmacy

1. Alcohol is purified from the mixture of alcohol and water obtained from fermentation tank.
2. It is used for the separation of miscible liquids, such as, alcohol and water, acetone and water, chloroform and benzene.

#### 4. Steam Distillation (Distillation of two immiscible liquids)

Theory When two immiscible liquids are heated together, then the

mixture boils when the sum of the vapour pressure equals to the atmospheric pressure. The temperature at which mixture boils is lower than that of either of the liquids *i.e. the boiling point of the mixture is low* than that of the liquid with the lower boiling point. The temperatures which the mixture boils remains stationary until one of the liquids has been completely removed from the still.

Apparatus used for laboratory scale It consists of a "Steam Can" fitted with a cork having two holes.

Through one of the holes passes bent tube leading the steam to the flask containing the non-aqueous liquid.

This tube should reach almost to the bottom of the flask.

Another long tube which passes through the other hole reaches almost the bottom of the steam can. This tube acts as a safety tube, so that case the pressure inside

Moreover, when steam starts coming out from the safety tube, it indicates that the steam can is almost empty. The delivery tube carrying vapours from the flask is connected to the condenser to convert it into liquid which gets collected in the receiver. The non-aqueous liquid is placed in the flask. A small quantity of water is added to it. The steam can and the flask are heated simultaneously, so that a uniform flow of steam passes through the boiling mixture.

'Distillation is continued until all the non-aqueous liquid has distilled Over. The distillate is then collected in florentine receiver where oil is completely separated from water.

Applications in pharmacy

1. It is used for the preparation of volatile oils.
2. It is used to determine the percentage of volatile oil in the drug.
3. It is used for the distillation of volatile oil for its

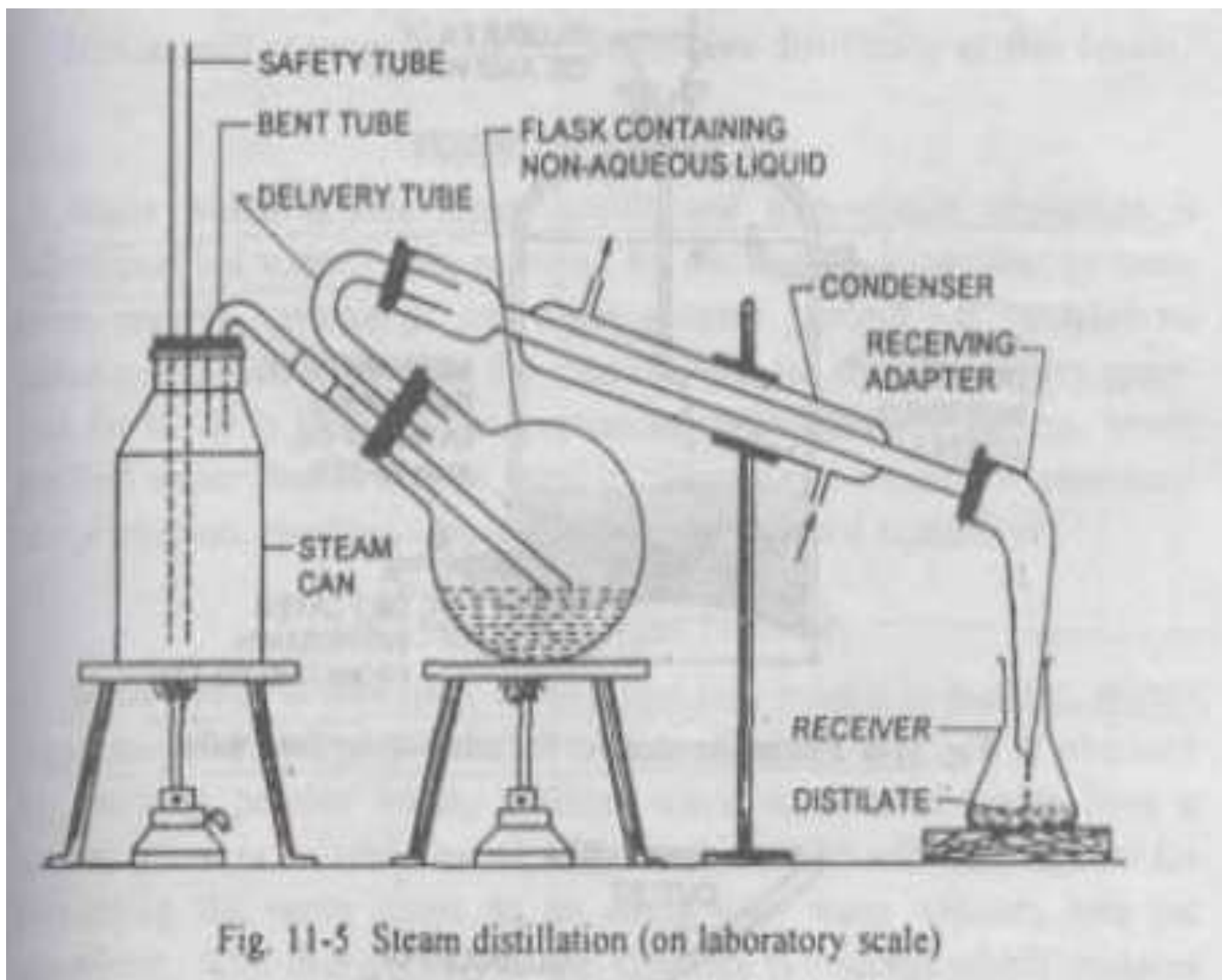


Fig. 11-5 Steam distillation (on laboratory scale)

## **FLORENTINE RECEIVERS**

It is used for the separation of oil and water. Florentine receivers are of two types:

Type I : used for separation of oil heavier than water.

Type II : used for separation of oil lighter than water.

The receiver used for oil heavier than water has two taps (Fig. 11-6}. The tap fitted near the bottom of vessel is used for collecting oil, whereas the tap fitted near the top .of the vessel is used for water to overflow.

The receiver used for oil lighter than water is fitted with syphon at the bottom which works when it gets filled with water (Fig. 11-1) whereas the tap fitted near the top is an outlet for the flow of oil.

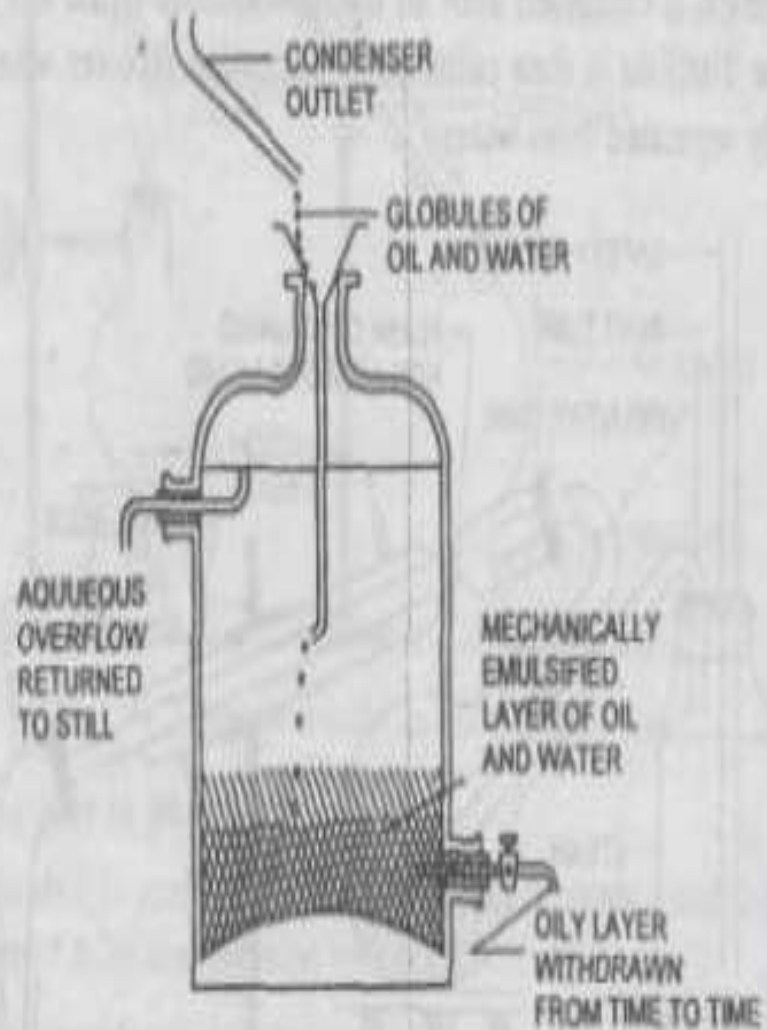


Fig. 11-6 Florentine receiver for oils heavier than water

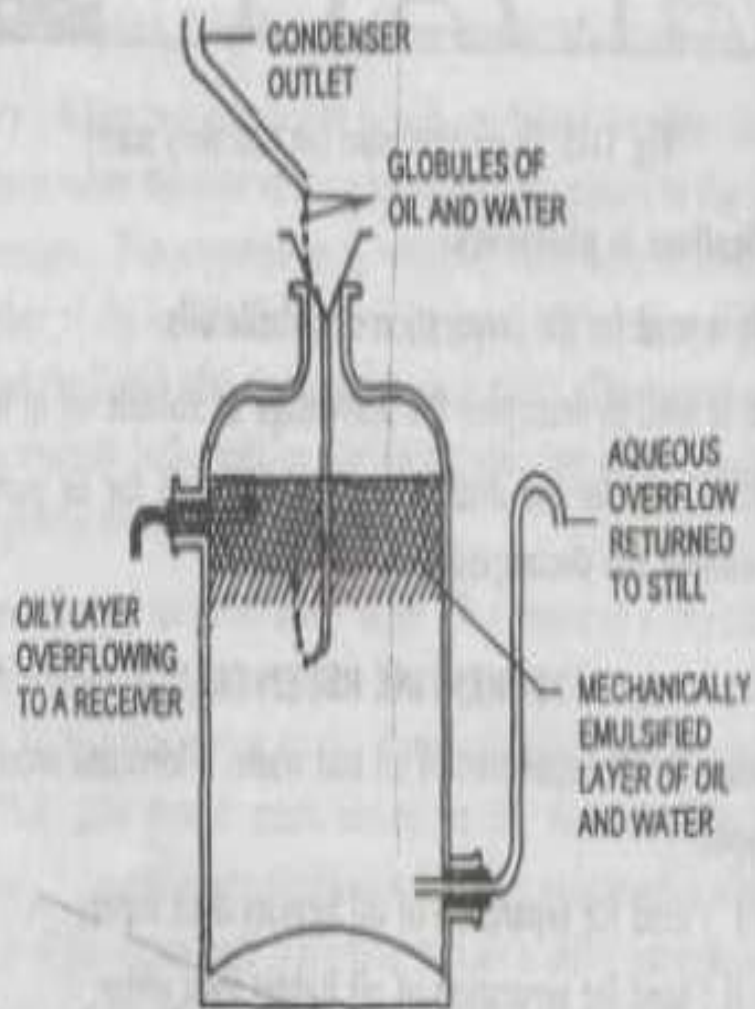


Fig. 11-7 Florentine receiver for oils lighter than water



## ***Destructive Distillation***

This is also known as Dry Distillation. The dried organic matter is

heated in the absence of air, in a suitable apparatus, until all the volatile substances are driven off and the residue is left behind. The residue is subjected to carbonisation.

Destructive distillation is mainly used in industry for obtaining many valuable products from wood and coal.

Destructive distillation of wood gives acetone, menthol, cresol, wood tar etc. while charcoal remains in the still.

Destructive distillation of coal gives burning gases the ammonia, and the coke remains in the still.

Destructive distillation of animal bones gives ammonia, amines and

hydrocarbons. Ichthammol is prepared by the destructive distillation of fish fossils.

## **PURIFIED WATER I.P.**

Water which is free from volatile and non-volatile impurities is

Called as purified water. It is prepared by distillation, ion-exchange treatment, reverse osmosis or any other suitable process. It contains no added substances and meets the requirements for chemical purity specified for it. It is liable to get contaminated by micro-organisms, hence purified water should not be used in preparations meant for parenteral administration. It should be stored in tightly closed containers.

## **WATER FOR INJECTION I.P.**

Water which is free from volatile and non-volatile impurities, microorganisms and pyrogens is called "Water for Injection". It is obtained by distilling potable water, purified water or distilled water from a neutral glass or suitable metal still fitted with an efficient device for preventing the water drops to go along with water

The first portion of the distillate is rejected which contains volatile impurities. The remainder is collected in suitable containers, previously rinsed with freshly distilled water and closed so as to avoid Contamination. It contains no added substances. Water for injection must meet the purity requirements stated under purified water. It need not be sterile but it should comply with the test for pyrogen. Water for injection is stored in tightly-closed neutral glass containers.

### **STERILE WATER FOR INJECTION**

it is water for injection which is sterilized and suitably packed, it contains no anti-microbial agent or other added substances. It has pH between 4.5 and 7.5. It must comply with the tests for sterility. It should also comply with the requirements of the tests for carbon dioxide, chloride, sulphate, nitrates and nitrites, ammonium, calcium and heavy metals. It must comply with the test for pyrogens. Sterile water for injection should be stored in single-dose containers not larger than of one liter in size.

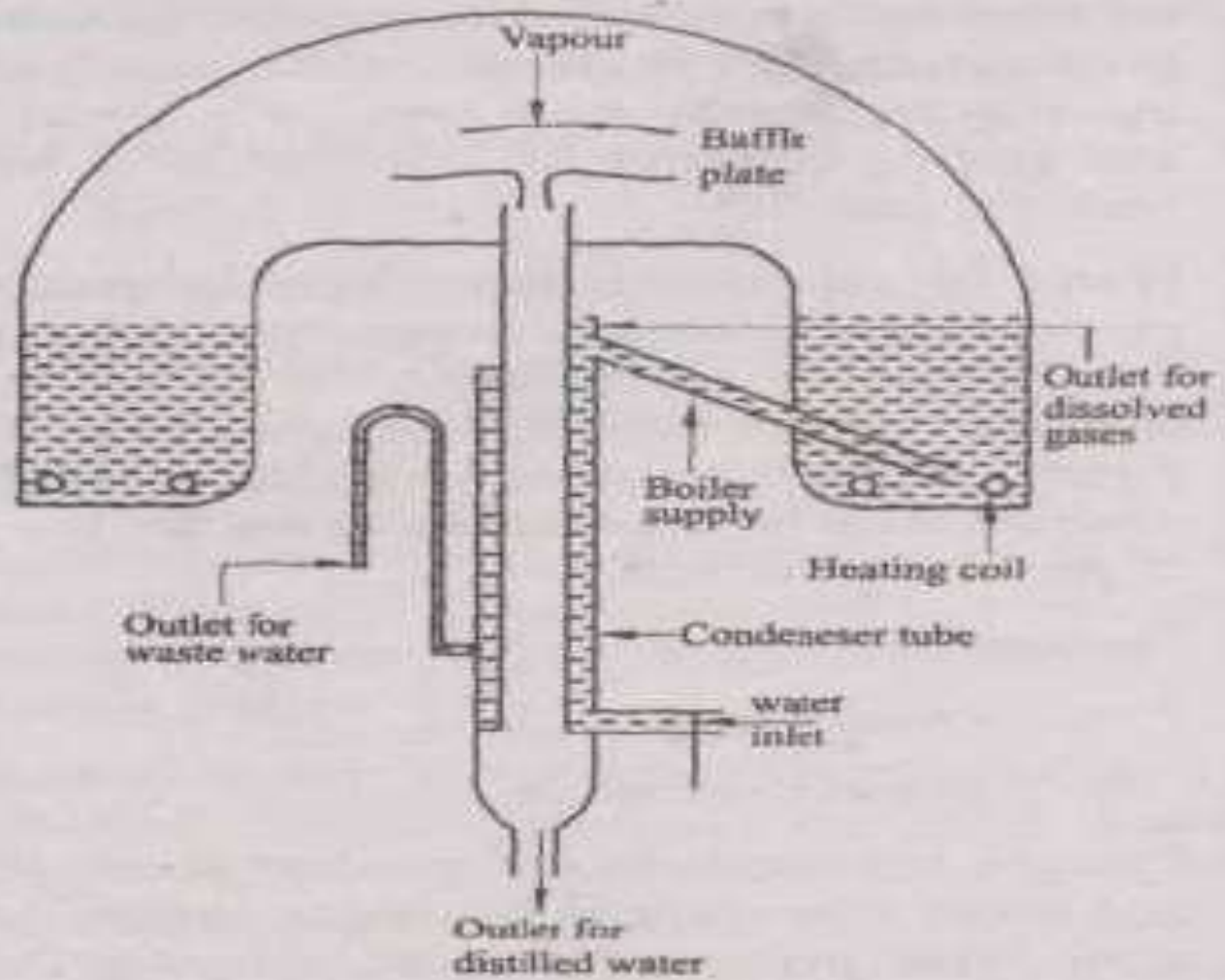


Fig. Still Head

## Preparation of Purified Water I.P. and Water for Injections I

by Distillation The potable water is used for the preparation of purified water. It contains— (1) Dissolved gases such as carbon-dioxide and ammonia (2) Dissolved salts and solids

This can be avoided by taking the following precautions: 1. By heating the feed water. This removes the dissolved gas. The solubility of gases decreases as the temperature is raised. For purpose of economy the water feeding the boiler should be through the condenser jacket for heating the water.

2. A constant level device is attached to the boiler to avoid excess concentration of salts. In spite of this, some of the solids will be deposited and it becomes necessary to de-scale the boiler after a certain period. Baffles are made of stainless steel. Baffles are provided over the top of the condenser tubes to avoid water drops getting mixed with the vapours. It is done to avoid carry-over of pyrogen and other water soluble materials in the droplet. The cooling water enters at the bottom of the condenser and is heated by the condensing vapours. The flow rate is adjusted in such a way that water gets heated at  $90^{\circ}$ - $95^{\circ}$ C before it enters the boiler. The top of the condenser jacket is open, so that gases from the water can escape into the atmosphere. A constant level device is fitted in such a way that the heated water free from

Evaporation	Distillation	Drying
<ol style="list-style-type: none"> <li>1. It means free secape of vapour from surface of liquid.</li> <li>2. It is observe at equilibrium between vapour pressure of liquid and atmospheric pressure.</li> <li>3. Not for thermolabile substance.</li> </ol>	<ol style="list-style-type: none"> <li>1. It is a process of conversion of liquid to vapour and reconvertng vapour in to liquid.</li> <li>2. It is done on the basis of difference of vapour pressure.</li> <li>3. It done under reduce pressure thermobile substance can be processed.</li> </ol>	<ol style="list-style-type: none"> <li>1. It is the process of removal of liquid from solid by vapourisation.</li> <li>2. It involves transfer of heat and mass.</li> <li>3. Freeze dryer can be used for thermolabile substance.</li> </ol>