

DISTILLATION

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DEFINITION

- “Distillation is defined as the separation of the components of a liquid mixture by a process involving vaporization and subsequent condensation at another place.”
- The distillation process involves 2 steps
 - (a) converting of liquid into vapour phase
 - (b) transferring the vapour to another phase and recovering the liquid by condensation.
- The feed liquid is known as distilland and the condensed liquid is known as distillate or condensate.
- If one component is volatile and others are non-volatile, it is possible to separate volatile components from non-volatile components by distillation. In such cases, distillation is considered as a separation or purification method.
- When heat is applied to a mixture, a more volatile liquid evaporates readily than the less volatile liquid.



APPLICATIONS:

- ❑ Separation of volatile oils- cloves(Eugenol comprises 72-90%, Vanilin, acetyl eugenol).
- ❑ Separation of drugs obtained from plant and animal sources- Vit. A from fish liver oil.
- ❑ Purification of organic solvents-absolute alcohol (100%).
- ❑ Purification of drugs obtained from chemical process.
- ❑ Refining of petroleum products- Petroleum ether 60,80.
- ❑ Recovery of solvents- synthesis.

GENERAL EQUIPMENT FOR DISTILLATION:

STILL :

- It is a vaporizing chamber and used to place the material to be distilled.
- The still is heated by a suitable means (ex-steam) for vaporization of the volatile constituents.
- On laboratory scale round bottom flasks made of glass are used so that the progress of the distillation can be noticed.
- A condenser is attached to the still using appropriate joints. A trap is inserted between distillation flask and condenser.

CONDENSER :

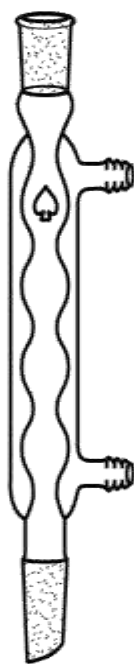
- Used to condense the vapour
- It is kept cold by circulating water/air through jacket. Types:
 - Single-surface condensers
 - Straight Tube
 - Bulb type
 - Spiral
 - Coiled type
 - Double-surface condensers
 - Multi-tubular condensers
- The condenser is connected to a receiver through a suitable adapter.

GENERAL EQUIPMENT FOR DISTILLATION:

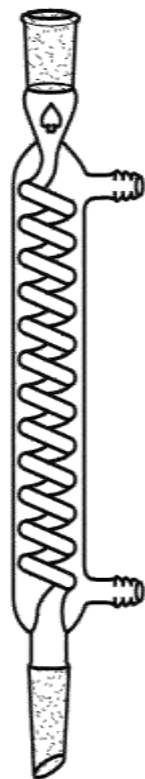
CONDENSER :



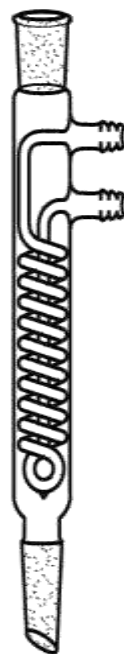
Liebig
Straight
Tube
type



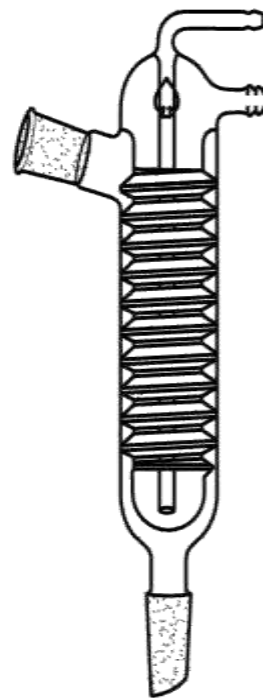
Allihn
Bulb type



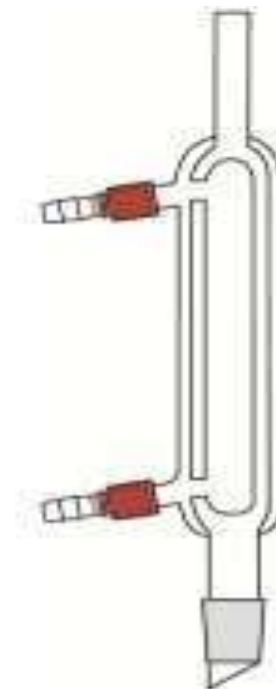
Graham
Spiral type



Dimroth
Coiled type



Friedrichs
Cold finger,
Spiral type



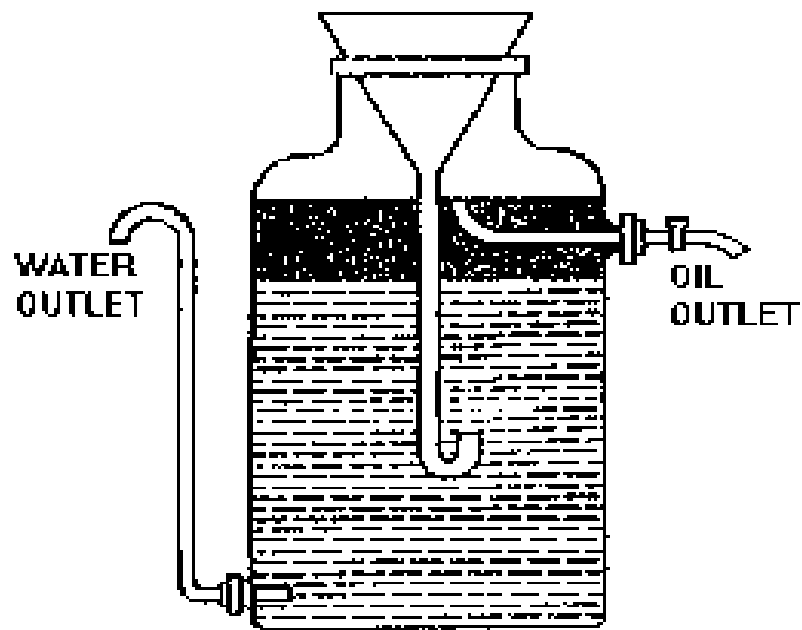
Lucas's
Double Surface

RECEIVER :

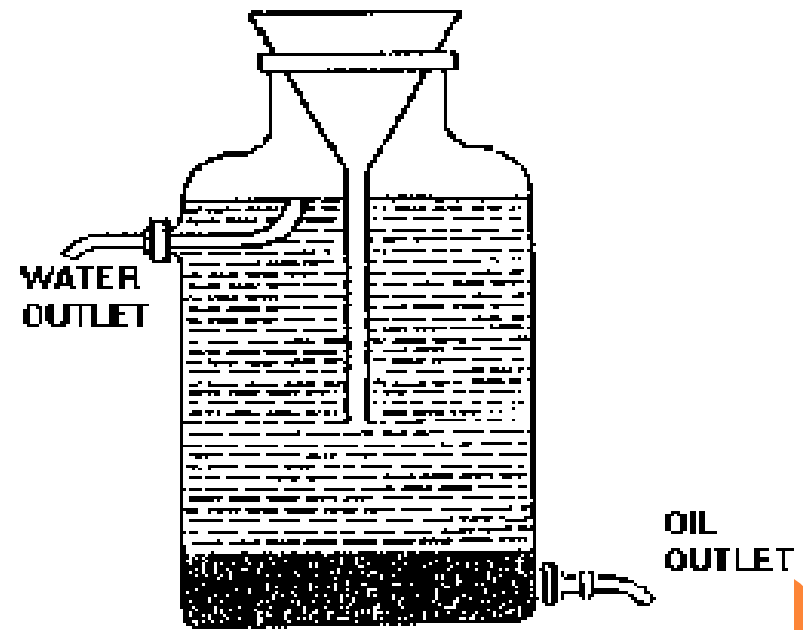
- It is used to collect the distillate.
- It may be a simple flask.
- It immersed in ice-bath to minimize loss of volatilization.
- Florentine receivers are used for the separation of oil and water.

Types of Florentine receivers :

- Type-I :- for separation of oil heavier than water.
- Type-II :- for separation of oil lighter than water.



FOR OILS LIGHTER
THAN WATER



FOR OILS HEAVIER
THAN WATER

CLASSIFICATION OF DISTILLATION METHODS

- I. Simple Distillation (**Differential distillation**)
- II. Flash Distillation (**Equilibrium distillation**)
- III. Fractional Distillation (**Rectification**)
- IV. Azeotropic and extractive Distillation
- V. Vacuum distillation (**distillation under reduced pressure**)
- VI. Steam Distillation
- VII. Molecular Distillation (**Evaporation distillation or short path distillation.**)
- VIII. Destructive Distillation
- IX. Compression Distillation

1. SIMPLE DISTILLATION

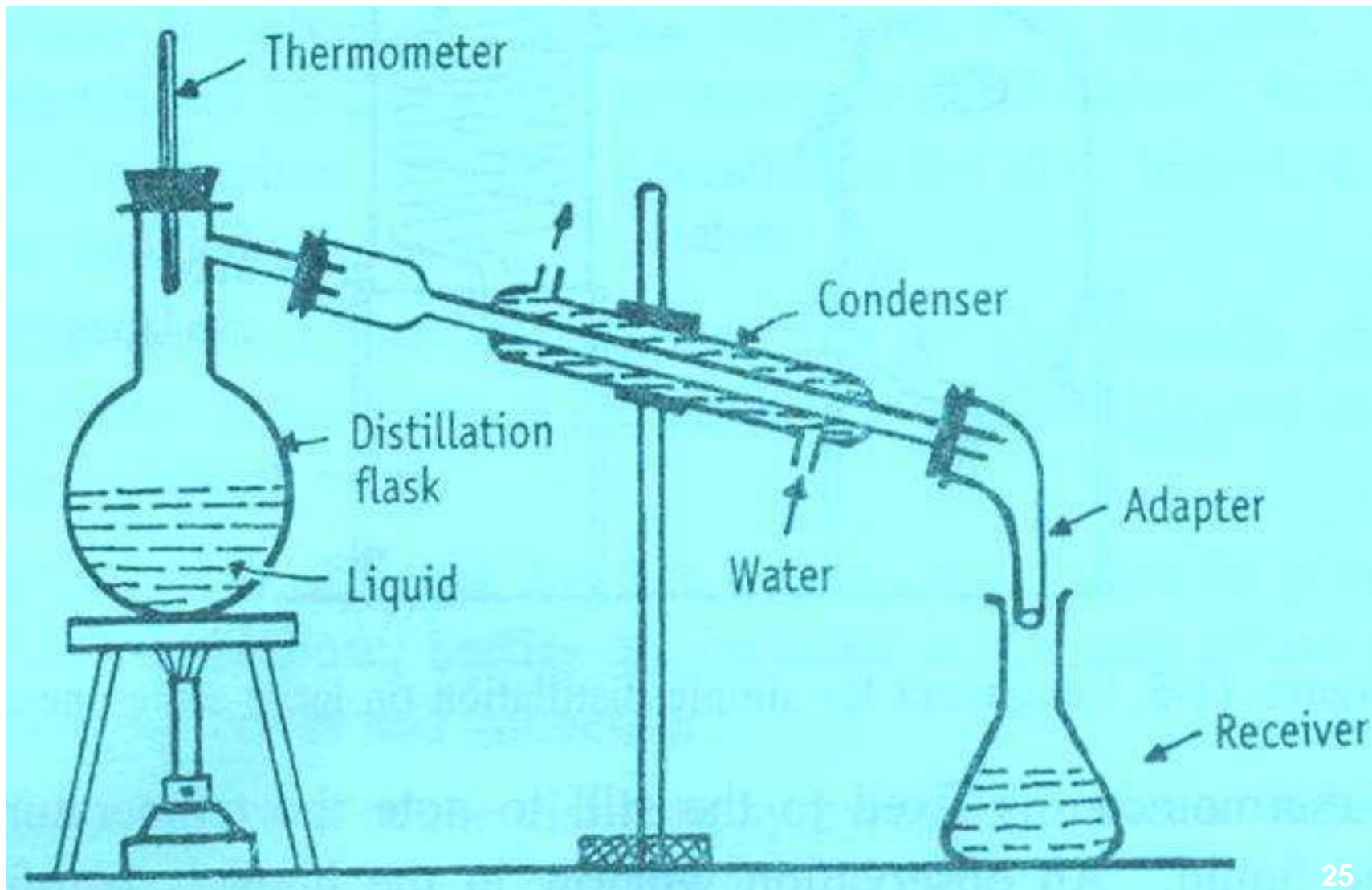
- Simple distillation is a process of converting a single constituent from a liquid (or mixture) into its vapour, transferring the vapour to another place and recovering the liquid by condensing the vapour, usually by allowing it to come in contact with a cold surface.
- This process is known differential distillation, as distillation is based on the differences in volatilities and vapour pressures of the components in the mixture. This method requires simple apparatus.

Principle:

- Liquid boils when its vapour pressure is equal to atmospheric pressure. Simple distillation is conducted at its boiling point.
- The higher the relative volatility of a liquid, the better is the separation by simple distillation. Heat is supplied to the liquid so that it boils. The resulting vapour is transferred to a different place and condensed.

CONSTRUCTION:

- It consists of a distillation flask with a side arm sloping downwards.
- Condenser is fitted into the side arm by means of a cork.
- The condenser is usually water condenser, i.e., jacketed for circulation of water.
- The condenser is connected to a receiver flask using an adapter with ground glass joints.
- On a laboratory scale, the whole apparatus is made of glass.



WORKING

- The liquid to be distilled is filled into the flask to one-half to two-third of its volume. Bumping is avoided by adding small pieces of porcelain or porous pot before distillation.
- A thermometer is inserted into the cork and fixed to the flask. The thermometer bulb must be just below the level of the side arm. Water is circulated through the jacket of the condenser. The contents are heated gradually.
- The liquid begins to boil after some time. The vapour begins to rise up and passes down the side arm into the condenser. The temperature rises rapidly and reaches a constant value.
- The **temperature of the distillate is noted down**, which is **equal to the boiling point of the liquid**. The vapour is condensed and collected into the receiver.
- The **flame is adjusted** so that the distillate is collected at the rate of one to two drops per second. Distillation should be continued **until a small volume of liquid remains in the flask**.

Applications:

- For the preparation of **distilled water and water for injection**. Volatile and aromatic waters are prepared.
- Organic solvents are purified.
- A few official compounds are prepared by distillation. Examples are spirit of nitrous ether and aromatic spirit of ammonia.
- Non-volatile solids are separated from volatile liquids.