



# **ORGANIC CHEMISTRY- SOME BASIC PRINCIPLES AND TECHNIQUES**



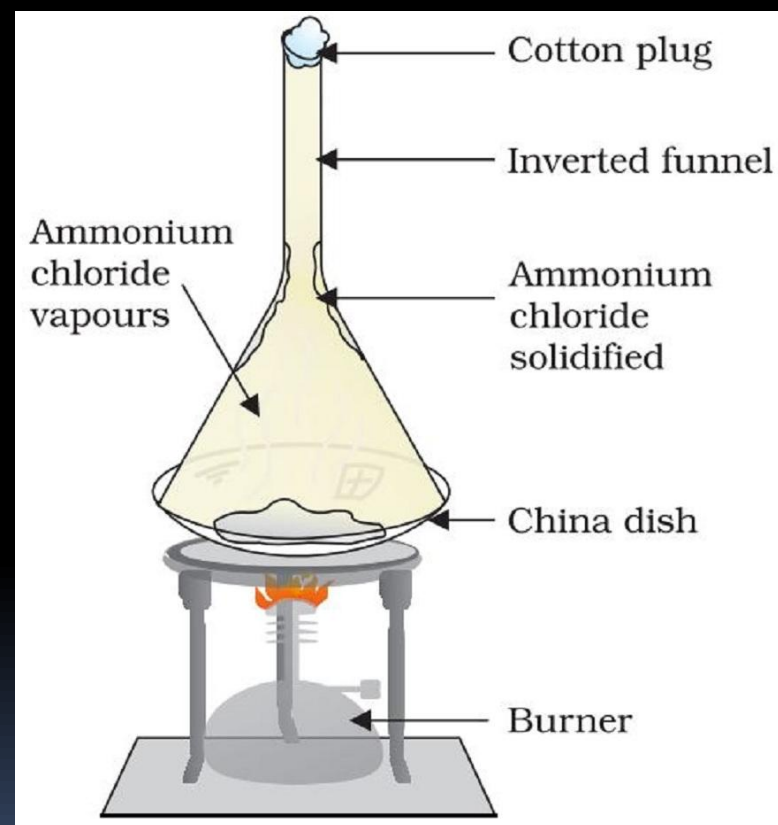
## **Purification Of Organic Compounds**

# METHODS OF PURIFICATION OF ORGANIC COMPOUNDS

- Regardless of whether, the substance has been extracted naturally or is synthesized in laboratory, the purification of an organic compound is necessary.
- Various methods employed for the purification of an organic compound depends upon the nature of substance being purified, and the impurity present in it.
- Various techniques used for Purification are-
  1. Sublimation
  2. Crystallisation
  3. Distillation
  4. Differential Extraction
  5. Chromatography

# SUBLIMATION

- It involves the conversion of a solid substance to gaseous vapours without passing through the intervening liquid state.
- It is used to separate sublimable compound from non- sublimable impurities.
- PROCEDURE: The substance is heated in a china dish above which an inverted funnel is kept to collect the sublimable compounds. The funnel is kept cool so as to hasten the process of solidification. Vapours of the substance solidify on the funnel.

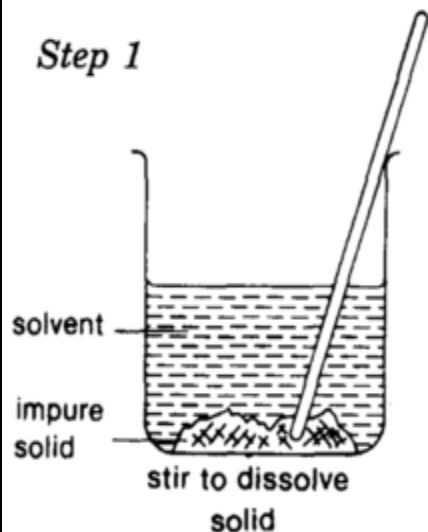


# CRYSTALLISATION

- Used for the purification of solid organic compounds.
- PRINCIPLE: It is based on the difference in the solubilities of the compound and the impurities in a suitable solvent.
- PROCEDURE: The impure compound is dissolved in a solvent in which it is sparingly soluble at room temperature but quite soluble at higher temperature to obtain a saturated solution. The hot solution is then filtered to remove any undissolved impurities. Upon cooling the solution, pure compound crystallizes out and is removed by filtration and the remaining filtrate contains impurities.
- CHOICE OF SOLVENT:
  - (a) The organic compound should be more soluble in it at higher temperature than at room temperature.
  - (b) It does not react chemically with the compound to be crystallized.
  - (c) Commonly used solvents are: water, alcohol, ether, chloroform, carbon-tetrachloride, acetone, benzene, petroleum ether etc.

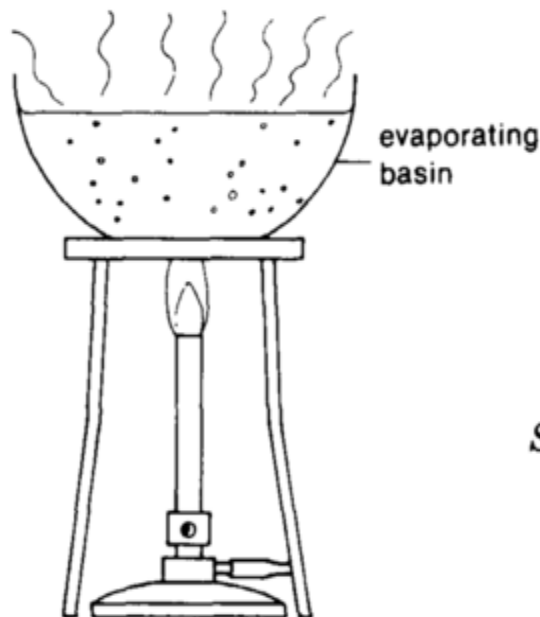
# SETUP FOR CRYSTALLISATION

*Step 1*



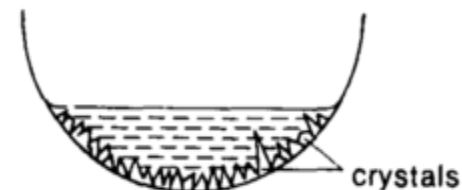
The impure solid is dissolved in a solvent.

*Step 2*



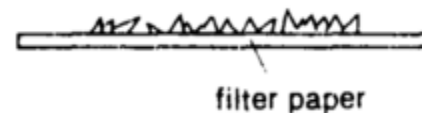
The solution is heated to evaporate most of the solvent.

*Step 3*



The hot solution is allowed to cool. The solid appears as pure crystals.

*Step 4*



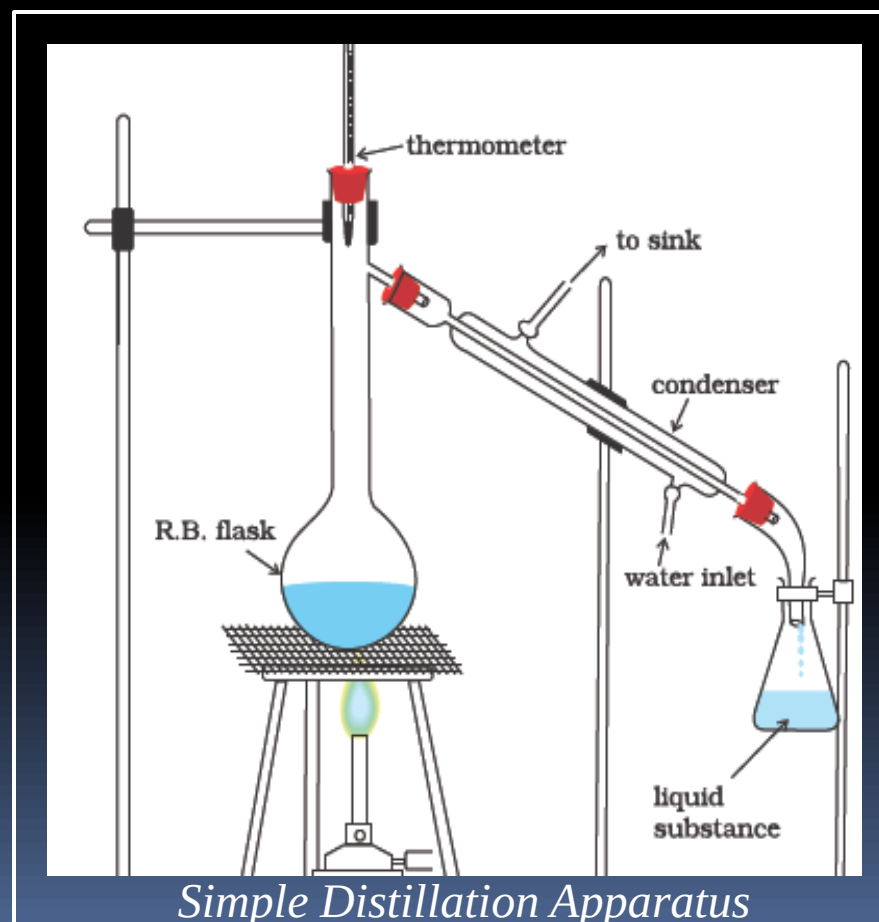
The cold solution is poured off to obtain the crystals. The crystals may be dried by pressing them between sheets of filter paper.

# DISTILLATION

- It involves the conversion of a liquid into vapours by heating followed by condensation of the vapours by cooling to obtain it in liquid state.
- PRINCIPLE: It is based on the fact that liquids having different boiling points vaporize at different temperatures and the mixture of liquids thus, can be separated due to the difference in their boiling points.
- Distillation can be of the following types-
  - (a) Simple Distillation
  - (b) Fractional Distillation
  - (c) Distillation under Reduced pressure
  - (d) Steam Distillation

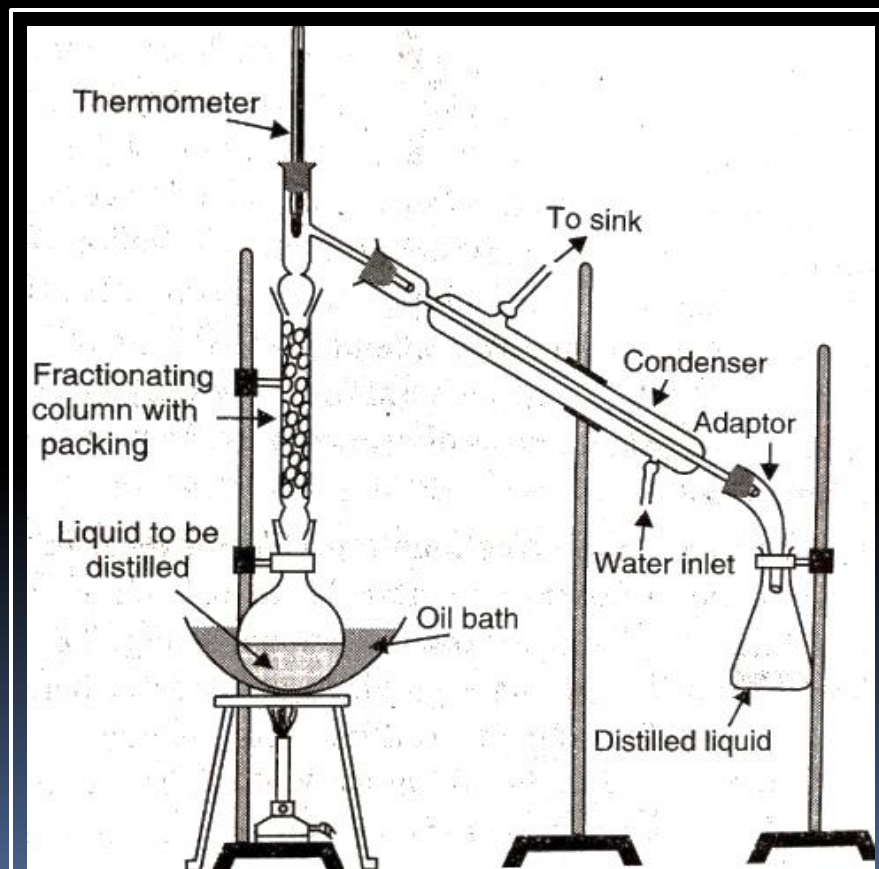
# SIMPLE DISTILLATION

- This method of distillation is used when the difference in boiling points of the two liquids is more than  $25^{\circ}\text{C}$ .
- Example-Mixture of Chloroform ( $60^{\circ}\text{C}$ ) & Aniline ( $189^{\circ}\text{C}$ )
- PROCEDURE: The liquid mixture is taken in a round bottom flask and heated carefully. On boiling, the vapours of lower boiling component are formed first, are condensed using a condenser and the liquid is collected in a receiver. The vapours of higher boiling component form later and the liquid can be collected separately



# FRACTIONAL DISTILLATION

- This method of distillation is used when the difference in boiling points of the two liquids is less than  $25^{\circ}\text{C}$ . The vapours of such liquids are formed within the same temperature range and are condensed simultaneously, thus, a fractionating column is fixed to the mouth of the RB.
- Example: mixture of Acetone ( $56^{\circ}\text{C}$ ) and Methyl Alcohol ( $65^{\circ}\text{C}$ ).
- PROCEDURE: The mixture to be purified is heated. The vapours rising up in the fractionating column become richer in low boiling component, pass through the condenser and the pure liquid (low boiling component) is collected in a receiver.



*Fractional Distillation Apparatus*

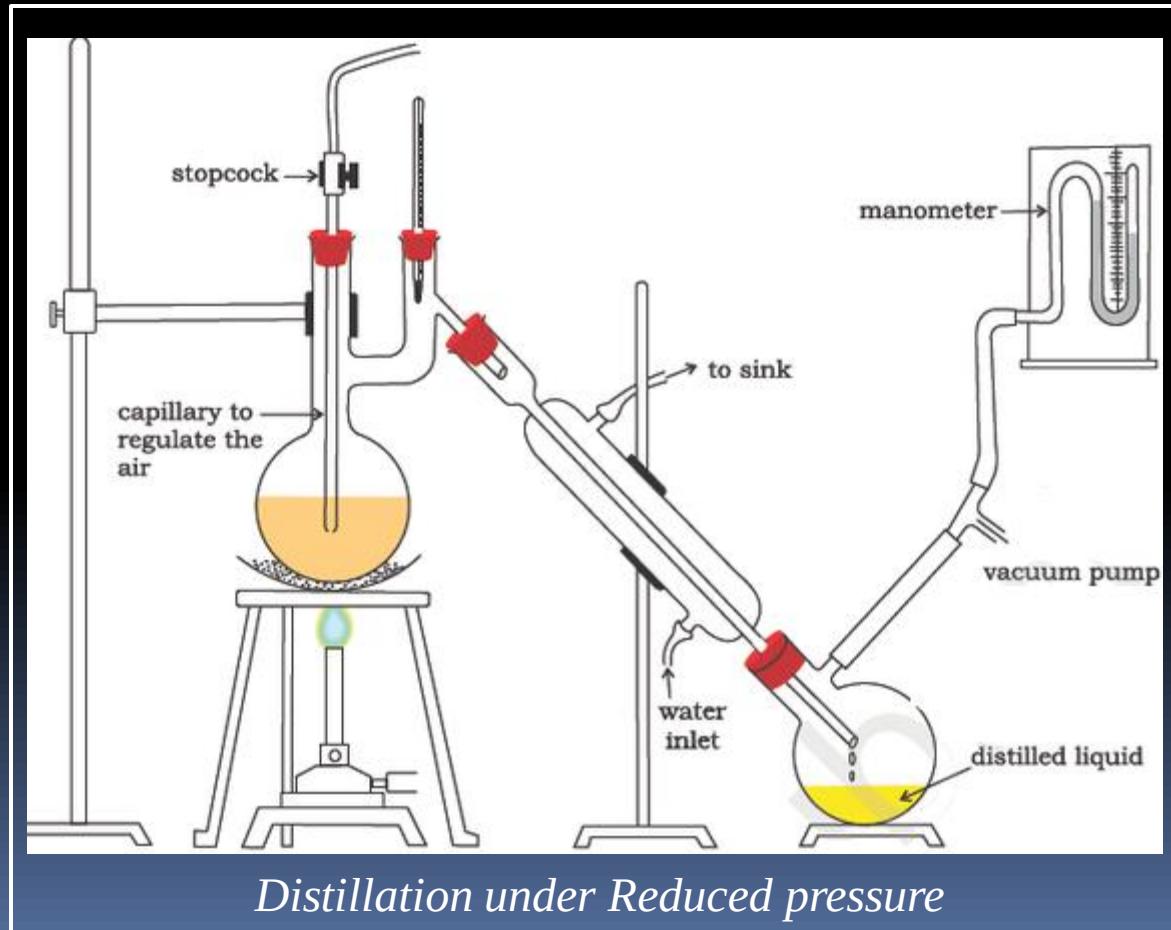
# DISTILLATION UNDER REDUCED PRESSURE

- It is used to purify liquids having very high boiling points and those, which decompose at or below their boiling points.
- Example- Glycerol

- PROCEDURE:

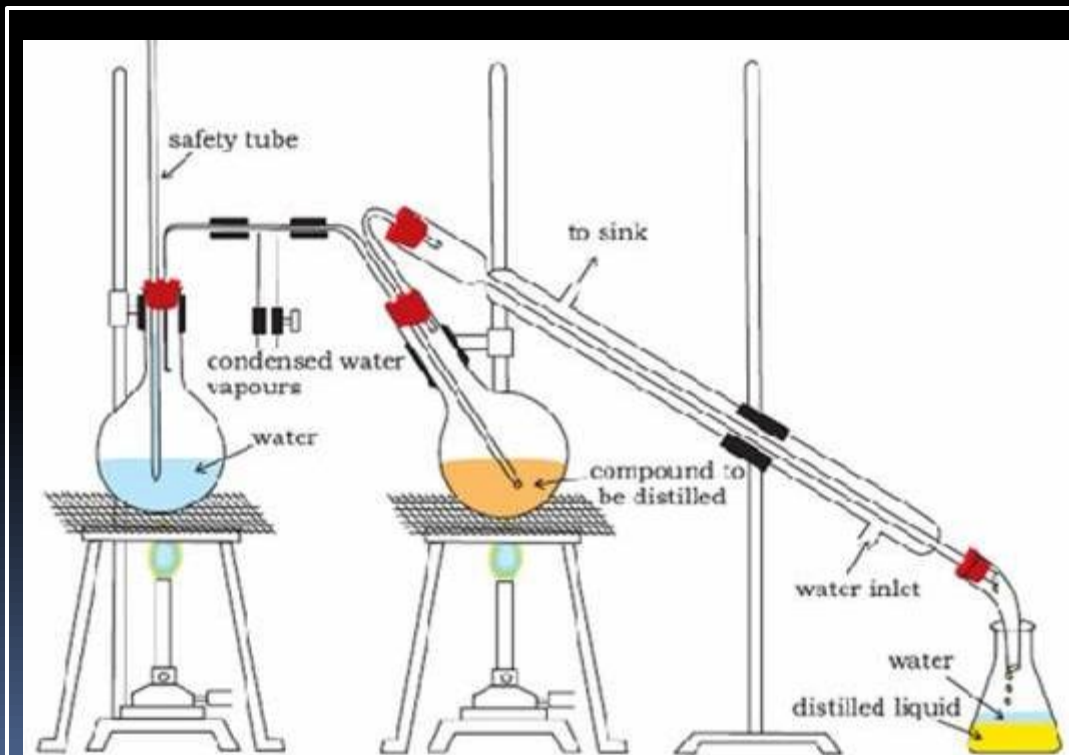
Since  $P \propto T$ , thus, the liquids will boil at a lesser temperature if they were distilled in an atmosphere having lower pressure.

This is achieved by using a vacuum pump. Thus, lowering the pressure will lower the boiling point of the liquid.



# STEAM DISTILLATION

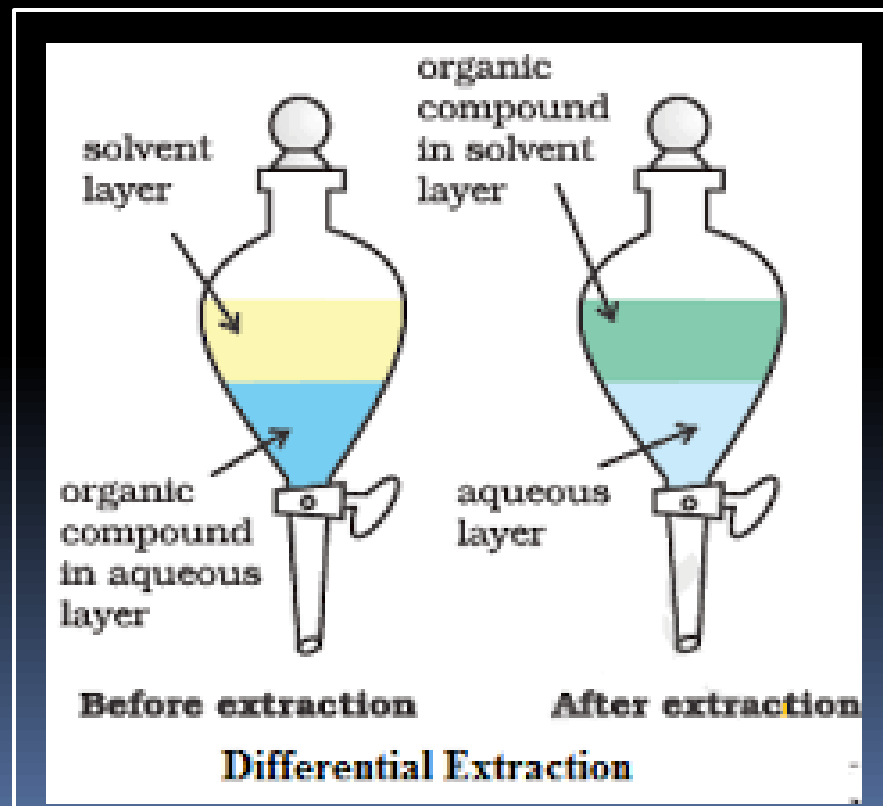
- This method is applicable for the separation and purification of those organic compounds which-
  - (a) are insoluble in water
  - (b) are volatile in steam
  - (c) possess a high vapour pressure (10-15 mm Hg) at 373 K and
  - (d) contain non-volatile impurities.
- Example : Aniline- Water mixture.
- Liquids will boil faster because of aqueous tension (vapour pressure of water) which helps in equalizing the atmospheric pressure.
- Total pressure = Aqueous tension + vapour pressure of liquid components



*Steam Distillation Apparatus*

# DIFFERENTIAL EXTRACTION

- It is used for the separation of an organic compound (solid or liquid) from its aqueous solution by shaking with a suitable organic solvent (e.g. ether, benzene, chloroform, carbon tetrachloride etc.) in which it is more soluble, in a separating funnel.
- A good solvent for extraction should follow the following two conditions:
  - (a) The substance to be extracted should be highly soluble in the solvent.
  - (b) After the extraction, the solvent should be easily separable from the compound.
- PROCEDURE: The immiscible liquids are taken in a separating funnel and left undisturbed. After a while, they separate out according to their specific gravities, with the heavier liquid at the bottom. Then they are later collected.



# CHROMATOGRAPHY

- It is a modern method used for the separation of mixtures into its components, purification of compounds and also to test the purity of compounds.
- The name chromatography is based on the *Greek* word '*chroma*' meaning colour as it was first used for the separation of coloured substances found in plants.
- In this technique, the mixture of substances is applied onto the stationary phase (solid mostly) and solvent (liquid or gas) is allowed to move slowly over the stationary phase which is known as mobile phase by which mixtures get separated into various components.
- PRINCIPLE: It is based on the difference in the rates at which the components of a mixture move through a stationary phase under the influence of some solvent or gas (called moving phase).
- Chromatography is of two types-
  - (a) Adsorption Chromatography
  - (b) Partition Chromatography

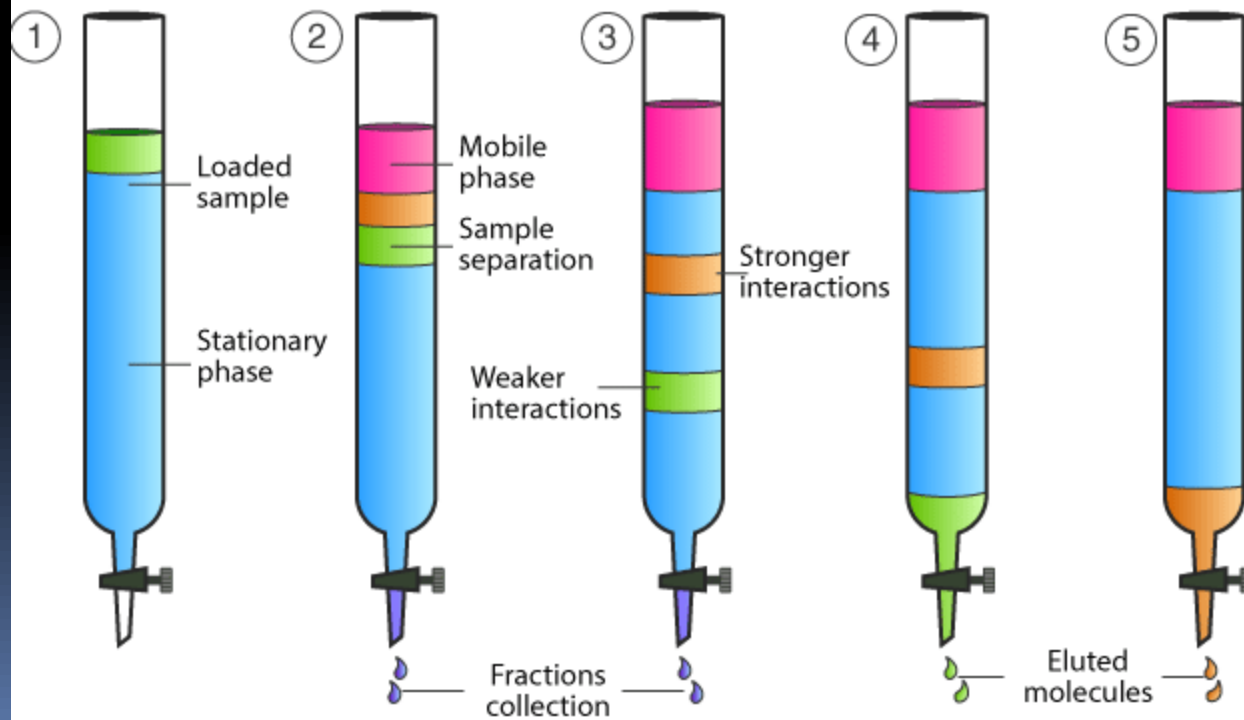
# ADSORPTION CHROMATOGRAPHY

- PRINCIPLE: It is based on the principle that different compounds are adsorbed on an adsorbent in varying degrees.
- The adsorbents used are generally silica gel or alumina. When a mobile phase (solvent) moves over the stationary phase (adsorbent), different constituents of the mixture get adsorbed at various distances over the fixed phase.
- Adsorption Chromatography can be classified into two types-
  - (a) Column Chromatography: Separation is done over a column of adsorbent (silica gel or alumina) packed in a glass column.
  - (b) Thin Layer Chromatography (TLC): It involves separation of the components of a mixture over a thin layer of adsorbent coated on a glass plate.

# COLUMN CHROMATOGRAPHY

- It involves separation of a mixture over a column of adsorbent (stationary phase) packed in a glass tube.
- PROCEDURE: The mixture is placed on the top of the adsorbent. A suitable solvent (liquid or mixture of liquids) is allowed to pass through the adsorbent. Depending upon the degree to which the components are adsorbed, the complete separation takes place.

- The component with most affinity with the stationary phase is adsorbed at the top, and so on.
- It is a type of Descending Chromatography.

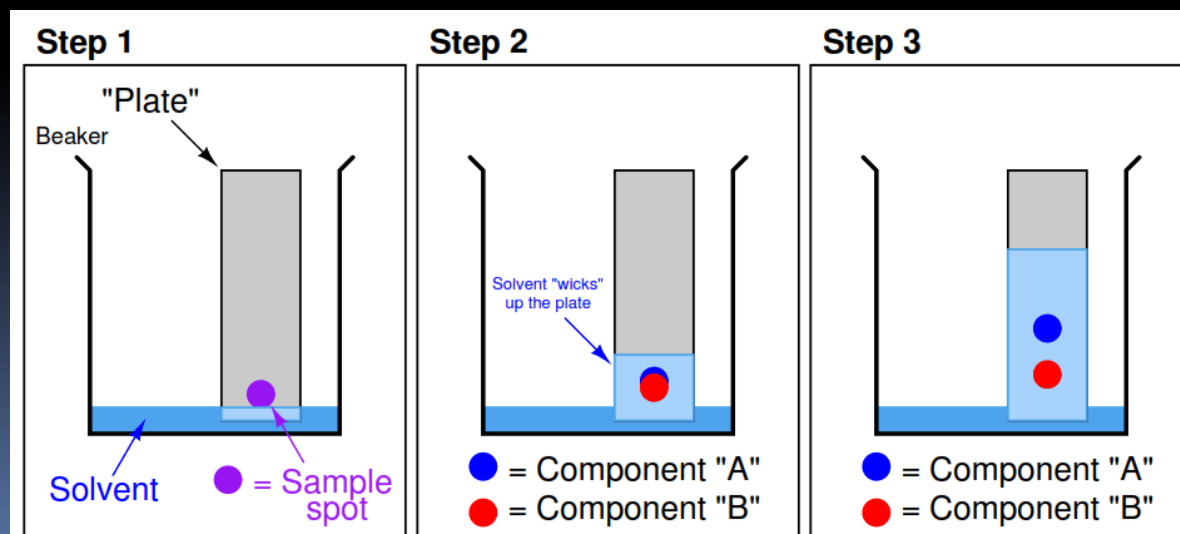


# THIN LAYER CHROMATOGRAPHY

- It involves separation of substances of a mixture over a thin layer of an adsorbent coated on glass plate.
- The plate is known as thin layer chromatography plate (TLC plate).
- PROCEDURE: The solution of the mixture to be separated is applied as a small spot about 2 cm above one end of the TLC plate. The glass plate is then placed in a closed jar containing the eluant. As the eluant rises up the plate, the components of the mixture move up along with the eluant to different distances depending on their degree of adsorption and separation takes place.

- It is a type of Ascending Chromatography.

- Retardation Factor/ Retention Factor ( $R_f$ ) can be calculated from TLC plate.



# PARTITION CHROMATOGRAPHY

- It is based on continuous differential partitioning of components of a mixture between stationary and mobile phases.
- Paper Chromatography is a type of Partition Chromatography. Chromatography paper contains water trapped in it, which acts as the stationary phase.

PROCEDURE: A strip of chromatography paper spotted at the base with the solution of the mixture is suspended in a suitable solvent or a mixture of solvents. This solvent acts as the mobile phase. The solvent rises up the paper by capillary action and flows over the spot. The paper selectively retains different components according to their differing partition in the two phases. The spots can be seen in UV light or with the help of a suitable spraying agent.

