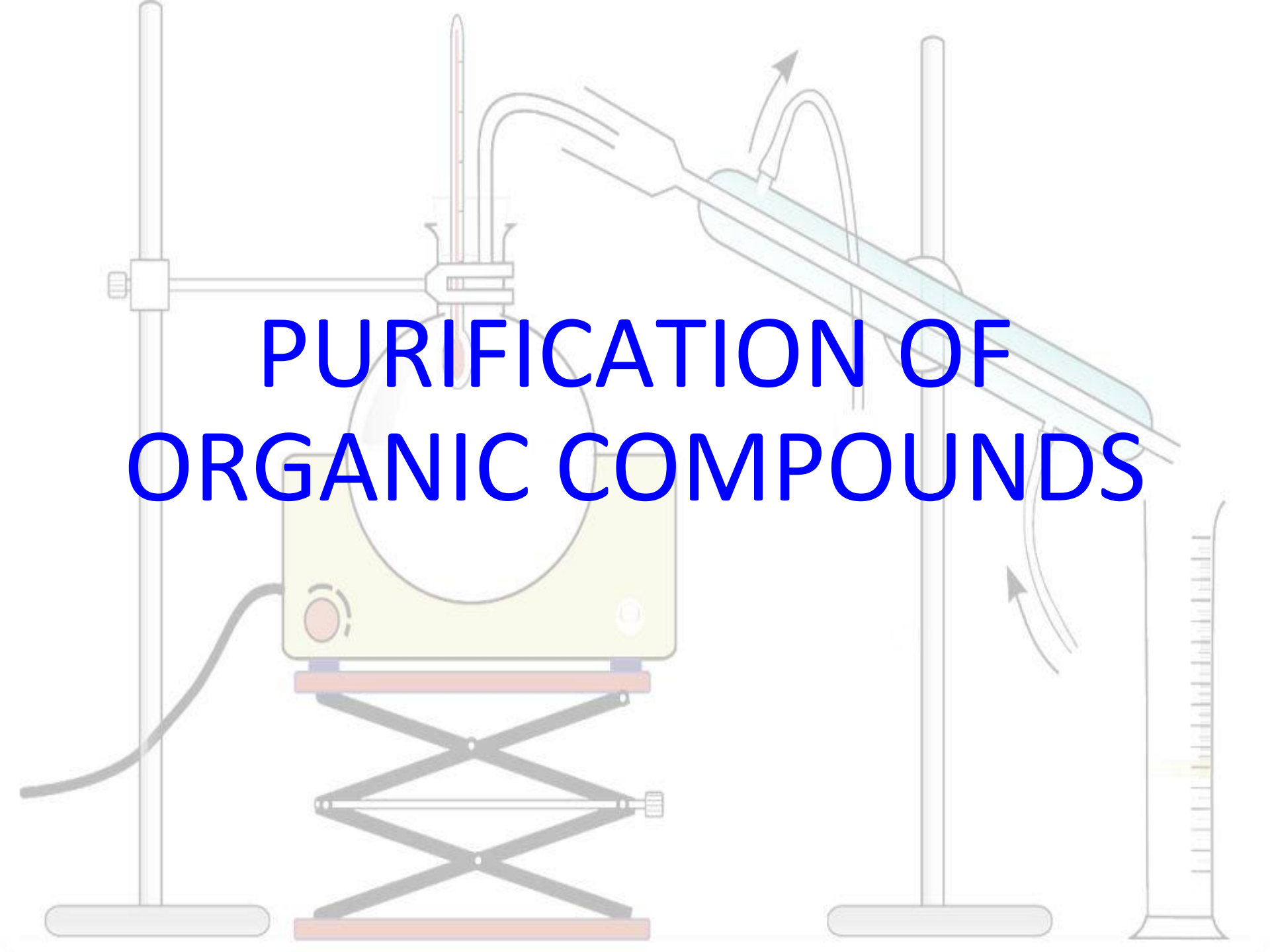


PURIFICATION OF ORGANIC COMPOUNDS

Purification of Organic Compounds

- These techniques include:
  1. Filtration
  2. Centrifugation
  3. Crystallization
  4. Solvent extraction
  5. Distillation

Purification of Organic Compounds

- These techniques include:

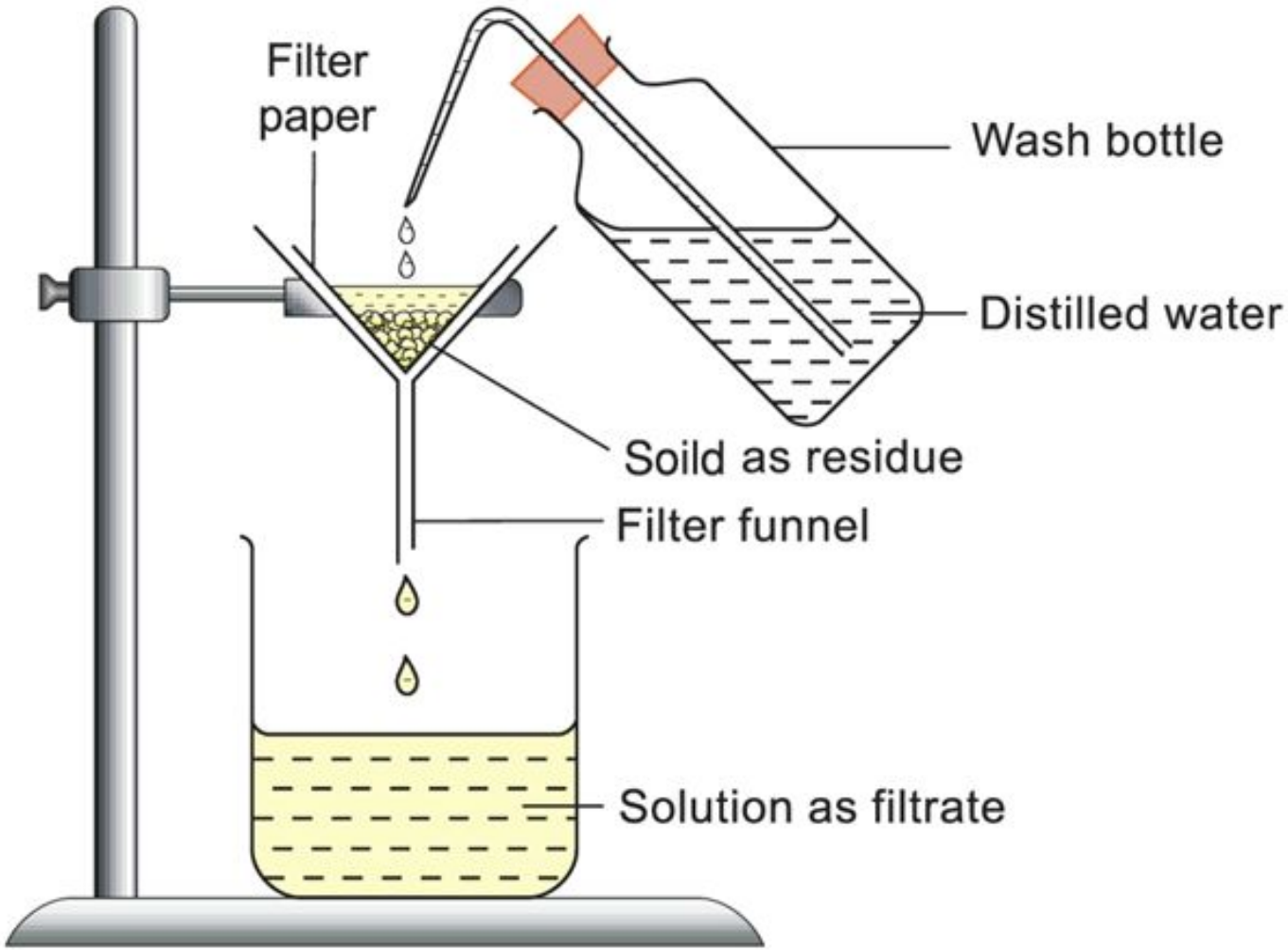
5. Fractional distillation
6. Sublimation
7. Chromatography
8. Steam distillation
9. Sublimation

By Using Filtration

Filtration

- To separate an insoluble solid from a liquid particularly when the solid is suspended throughout the liquid
- The solid/liquid mixture is called a suspension

Filtration



The laboratory set-up of filtration

Filtration

- There are many small holes in the filter paper

  ➔ allow very small particles of solvent and dissolved solutes to pass through as filtrate

- Larger insoluble particles are retained on the filter paper as residue

By Using Centrifugation

Centrifugation

- When there is only a small amount of suspension, or when much faster separation is required

➔ Centrifugation is often used instead of filtration

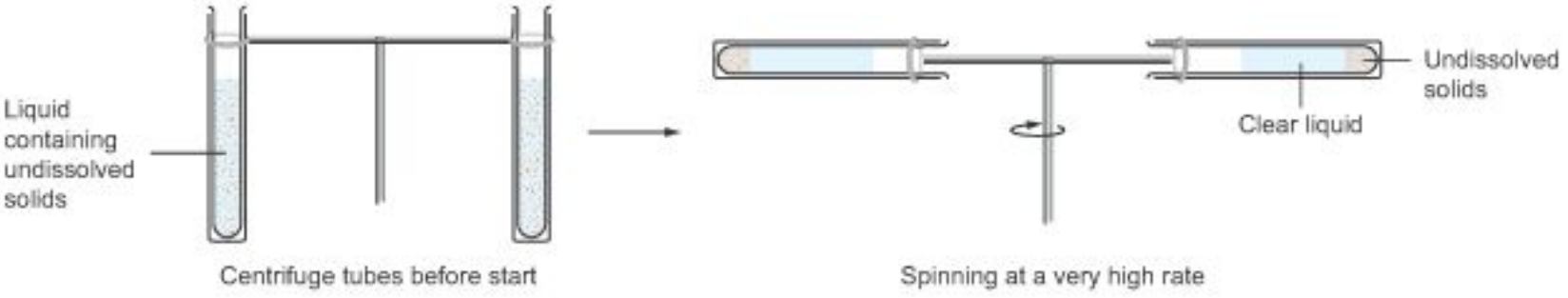
Centrifugation

- The liquid containing undissolved solids is put in a centrifuge tube
- The tubes are then put into the tube holders in a centrifuge

A centrifuge

Centrifugation

- The holders and tubes are spun around at a very high rate and are thrown outwards
- The denser solid is collected as a lump at the bottom of the tube with the clear liquid above



By Using Crystallisation

Crystallisation

- Crystals are solids that have

  ➔ a definite regular shape

  ➔ smooth flat faces and straight edges

- Crystallisation is the process of forming crystals

1. Crystallisation by Cooling a Hot Concentrated Solution

- To obtain crystals from an unsaturated aqueous solution

  ➔ the solution is gently heated to make it more concentrated

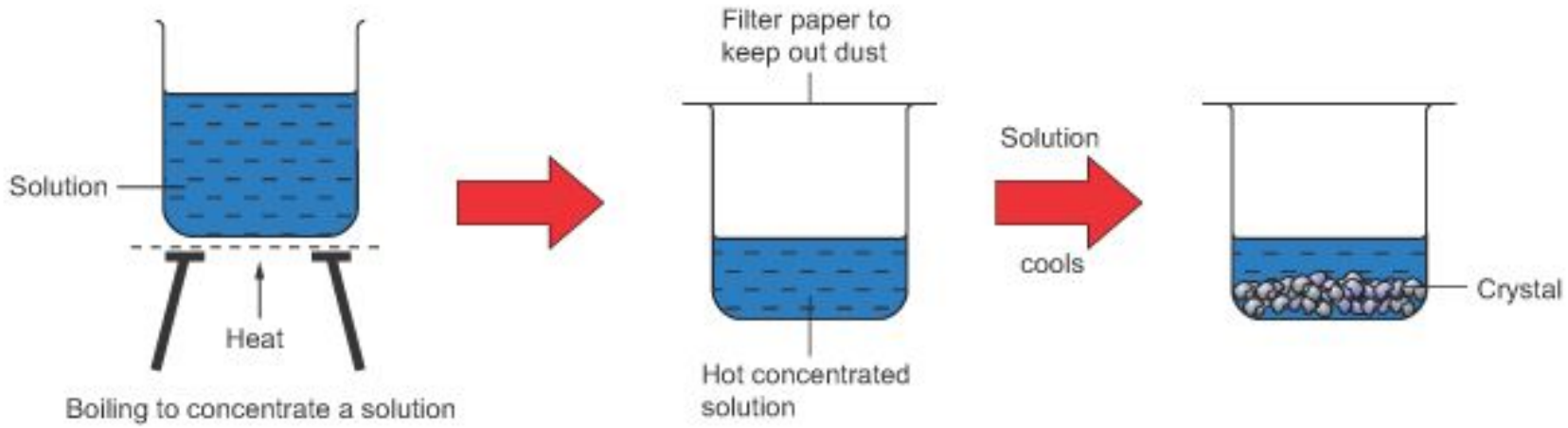
- After, the solution is allowed to cool at room conditions

1. Crystallisation by Cooling a Hot Concentrated Solution

- The solubilities of most solids increase with temperature
- When a hot concentrated solution is cooled

  ➔ the solution cannot hold all of the dissolved solutes
- The “excess” solute separates out as crystals

1. Crystallisation by Cooling a Hot Concentrated Solution



Crystallisation by cooling a hot concentrated solution

2. Crystallisation by Evaporating a Cold Solution at Room Temperature

- As the solvent in a solution evaporates,
- ➔ the remaining solution becomes more and more concentrated
- ➔ eventually the solution becomes saturated
- ➔ further evaporation causes crystallisation to occur

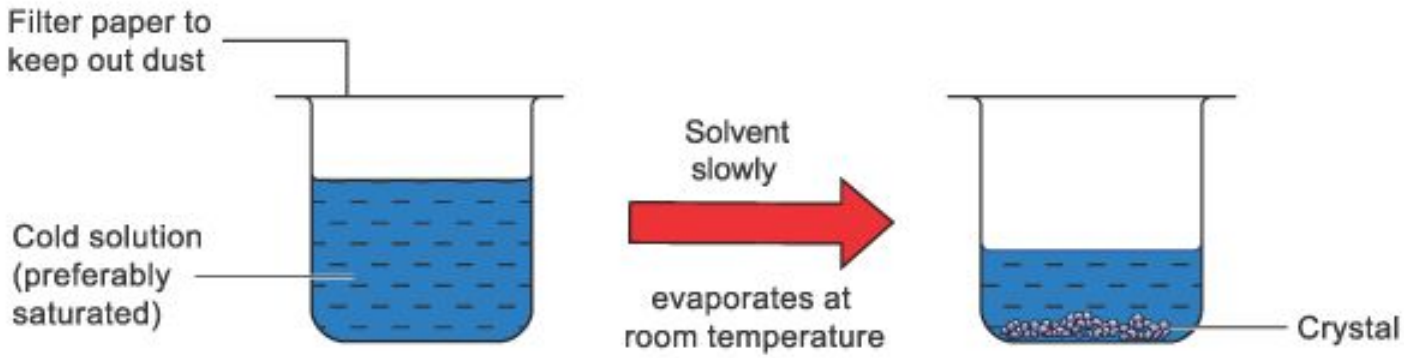
2. Crystallisation by Evaporating a Cold Solution at Room Temperature

- If a solution is allowed to stand at room temperature,

  → evaporation will be slow

- It may take days or even weeks for crystals to form

2. Crystallisation by Evaporating a Cold Solution at Room Temperature



Crystallisation by slow evaporation of a solution (preferably saturated) at room temperature

By Using Solvent Extraction

Solvent Extraction

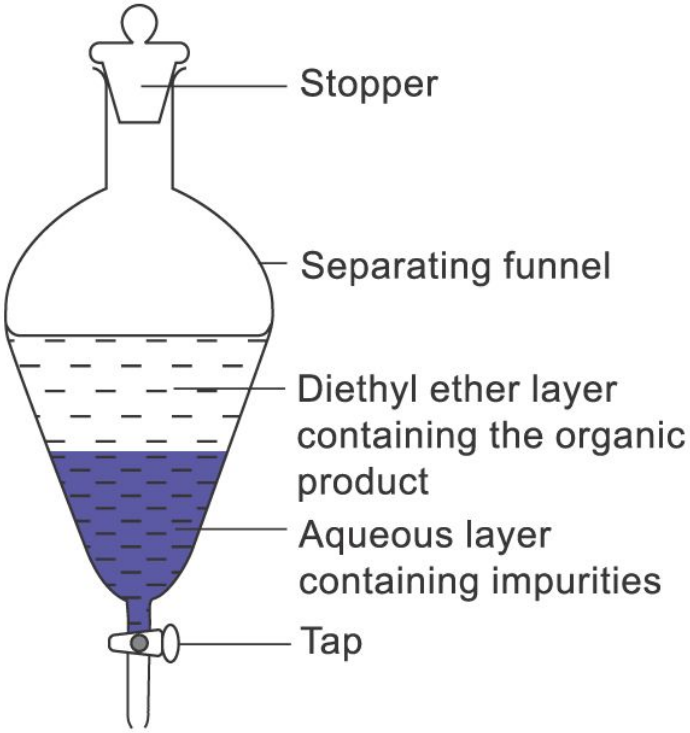
- Involves extracting a component from a mixture with a suitable solvent
- Water is the solvent used to extract salts from a mixture containing salts and sand
- Non-aqueous solvents (e.g. 1,1,1-trichloroethane and diethyl ether) can be used to extract organic products

Solvent Extraction

- Often involves the use of a separating funnel
- When an aqueous solution containing the organic product is shaken with diethyl ether in a separating funnel,

  ➔ the organic product dissolves into the ether layer

Solvent Extraction



The organic product in an aqueous solution can be extracted by solvent extraction using diethyl ether

Solvent Extraction

- The ether layer can be run off from the separating funnel and saved
- Another fresh portion of ether is shaken with the aqueous solution to extract any organic products remaining
- Repeated extraction will extract most of the organic product into the several portions of ether

Solvent Extraction

- Conducting the extraction with several small portions of ether is more efficient than extracting in a single batch with the whole volume of ether
- These several ether portions are combined and dried

  ➔ the ether is distilled off

  ➔ leaving behind the organic product

By Using Distillation

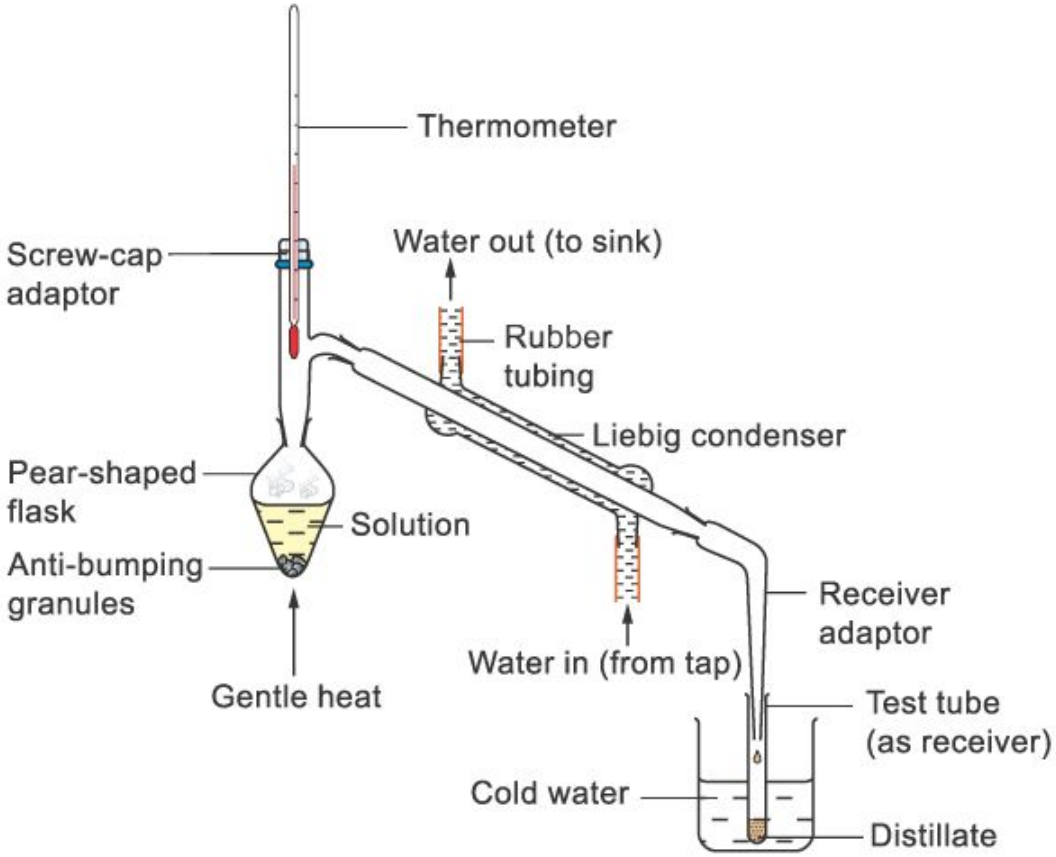
Distillation

- A method used to separate a solvent from a solution containing non-volatile solutes
- When a solution is boiled,

  ➔ only the solvent vaporizes

  ➔ the hot vapour formed condenses to liquid again on a cold surface
- The liquid collected is the distillate

Distillation



The laboratory set-up of distillation

Distillation

- Before the solution is heated,

  ➔ several pieces of anti-bumping granules are added into the flask

  ➔ prevent vigorous movement of the liquid called bumping to occur during heating

  ➔ make boiling smooth

Distillation

- If bumping occurs during distillation,

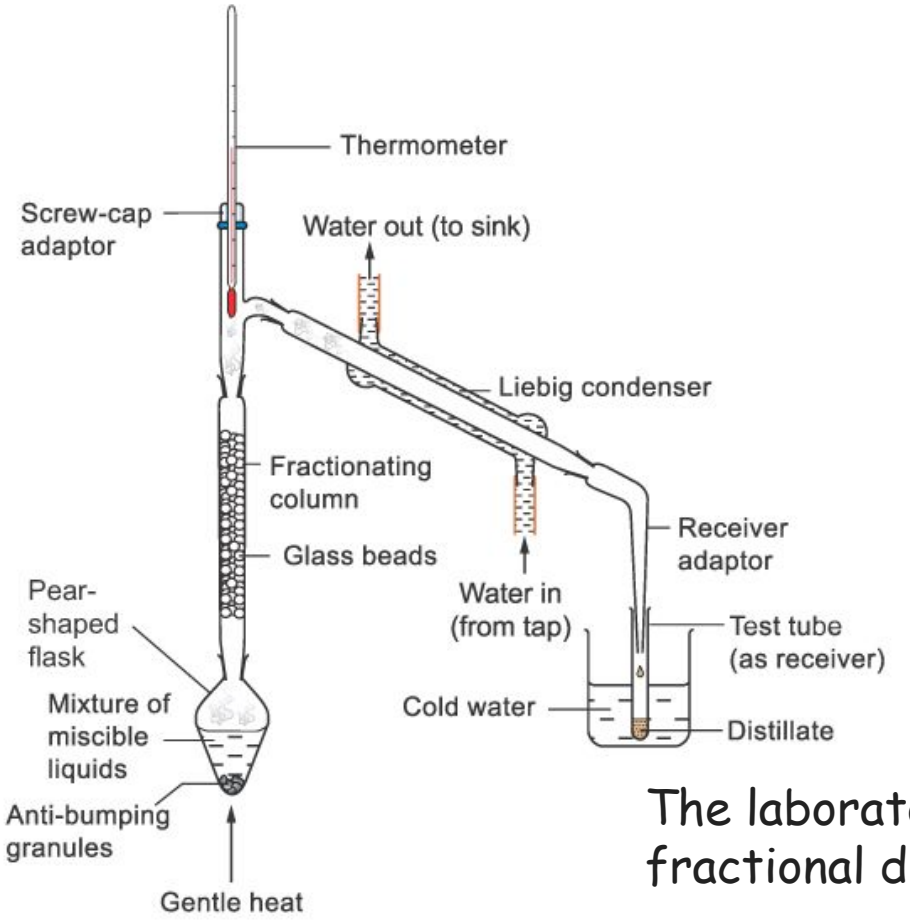
  → some solution (not yet vaporized) may spurt out into the collecting vessel

By Using Fractional Distillation

Fractional Distillation

- A method used to separate a mixture of two or more miscible liquids

Fractional Distillation



The laboratory set-up of fractional distillation

Fractional Distillation

- A fractionating column is attached vertically between the flask and the condenser

  ➔ a column packed with glass beads

  ➔ provide a large surface area for the repeated condensation and vaporization of the mixture to occur

Fractional Distillation

- The temperature of the escaping vapour is measured using a thermometer
- When the temperature reading becomes steady,

  ➔ the vapour with the lowest boiling point firstly comes out from the top of the column

Fractional Distillation

- When all of that liquid has distilled off,

  ➔ the temperature reading rises and becomes steady later on

  ➔ another liquid with a higher boiling point distills out

- Fractions with different boiling points can be collected separately

By Using Sublimation

Sublimation

- Sublimation is the direct change of

  ➔ a solid to vapour on heating, or

  ➔ a vapour to solid on cooling

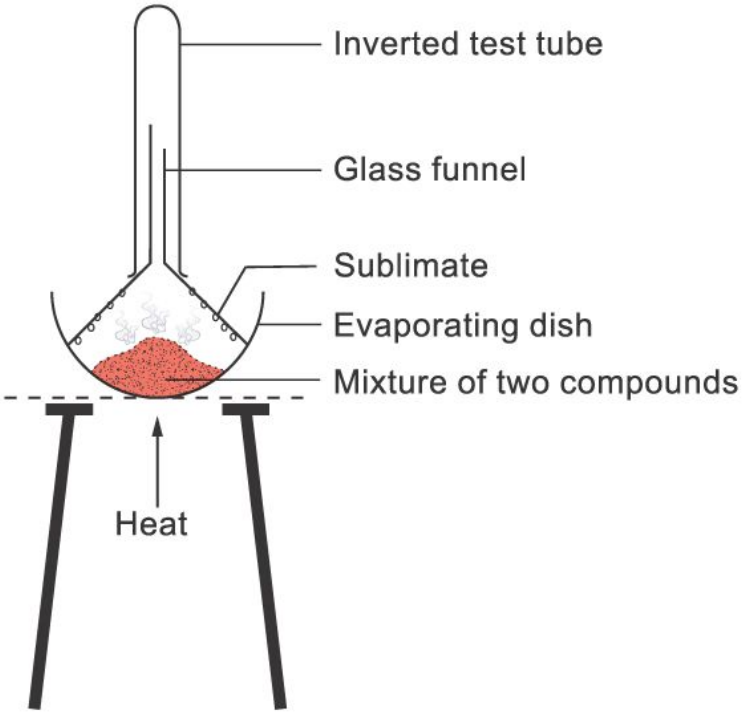
  ➔ without going through the liquid state

Sublimation

- A mixture of two compounds is heated in an evaporating dish
- One compound changes from solid to vapour directly

  ➔ The vapour changes back to solid on a cold surface
- The other compound is not affected by heating and remains in the evaporating dish

Sublimation



A mixture of two compounds can be separated by sublimation

3.DISTILLATION

Aim

To separate a solution of a solid in a liquid and for separating a solution of two liquids whose boiling points are different.

Principle

Distillation involves the conversion of a liquid into its vapors upon heating and then cooling the vapors back into the liquid. Depending on the difference in boiling points of liquids.

Types of distillation

- Simple Distillation
- Fractional Distillation
- Distillation Under Reduced Pressure or Vacuum Distillation
- Steam Distillation

SIMPLE DISTILLATION

Principle

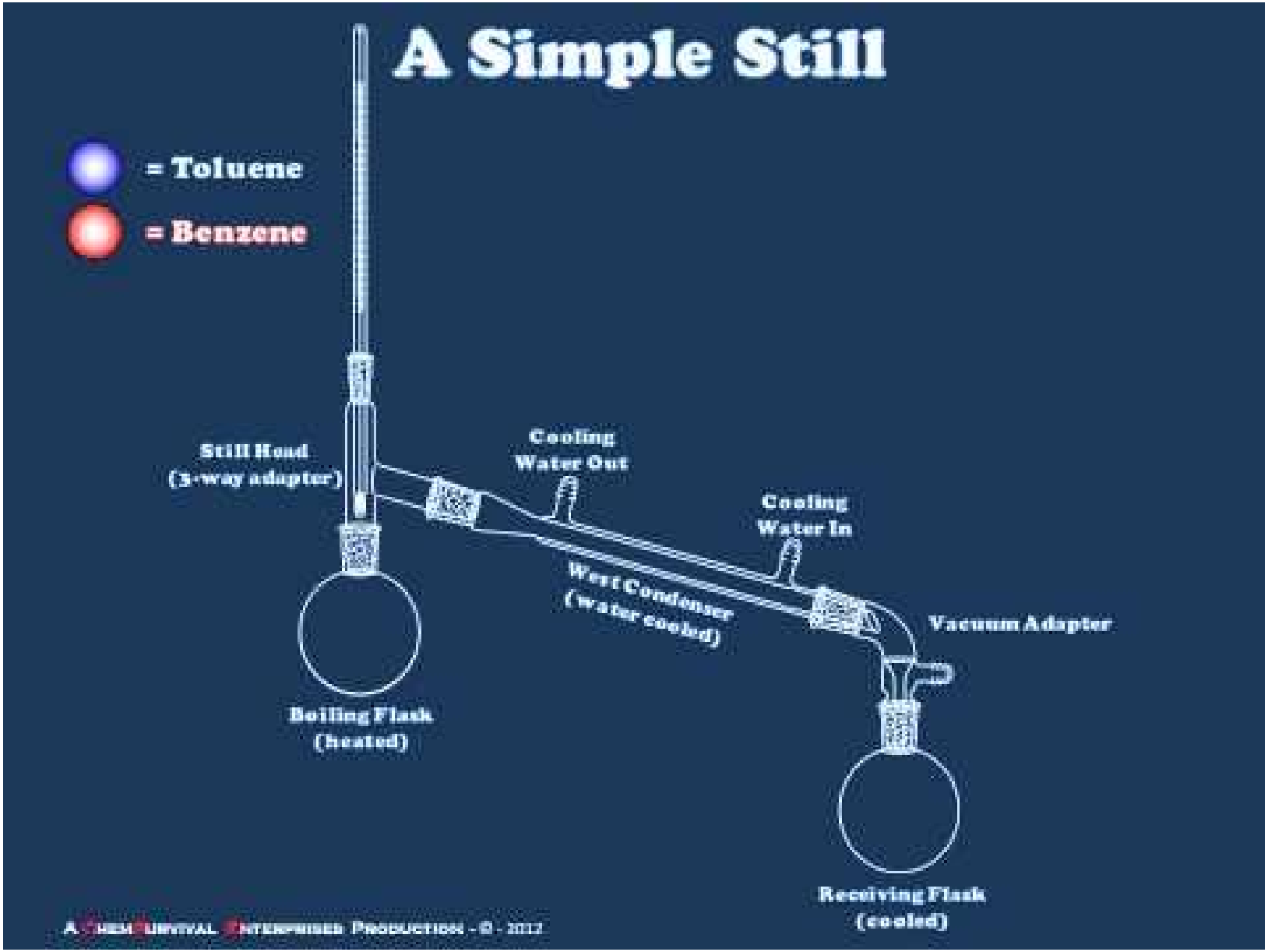
It is used for separating liquids having boiling points differing by 10-20 degrees. The liquid having the lower boiling point distills over first, and the other liquid component is left behind. In this process, vaporization and condensation occur side by side.

Process

Example

Simple distillation of a Cyclohexane- Toluene mixtures

How Simple Distillation Works ?



FRACTIONAL DISTILLATION

Principle

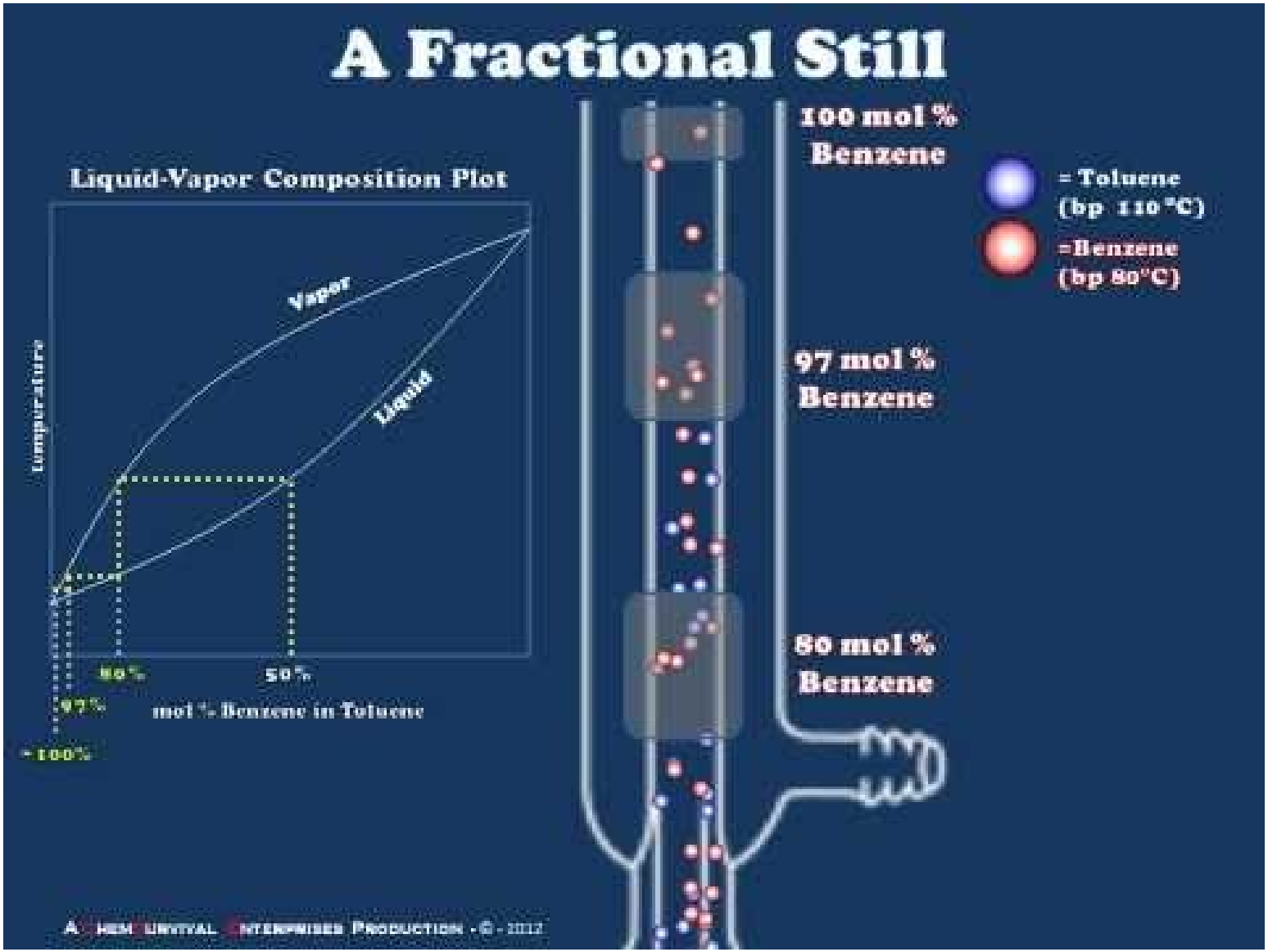
It is used for separating two liquids in any mixture, which have boiling points within a narrow range of temperatures. In such cases, simple distillation does not give complete separation and a modified version called fractional distillation is employed.

Process

Example

Fractional Distillation of a Cyclohexane- Toluene mixtures

How Fractional Distillation Works ?



STEAM DISTILLATION

Principle

This technique is used for separating/purifying liquids, which are immiscible with water, volatile in steam, & have high vapor pressure at the boiling temperature of water.

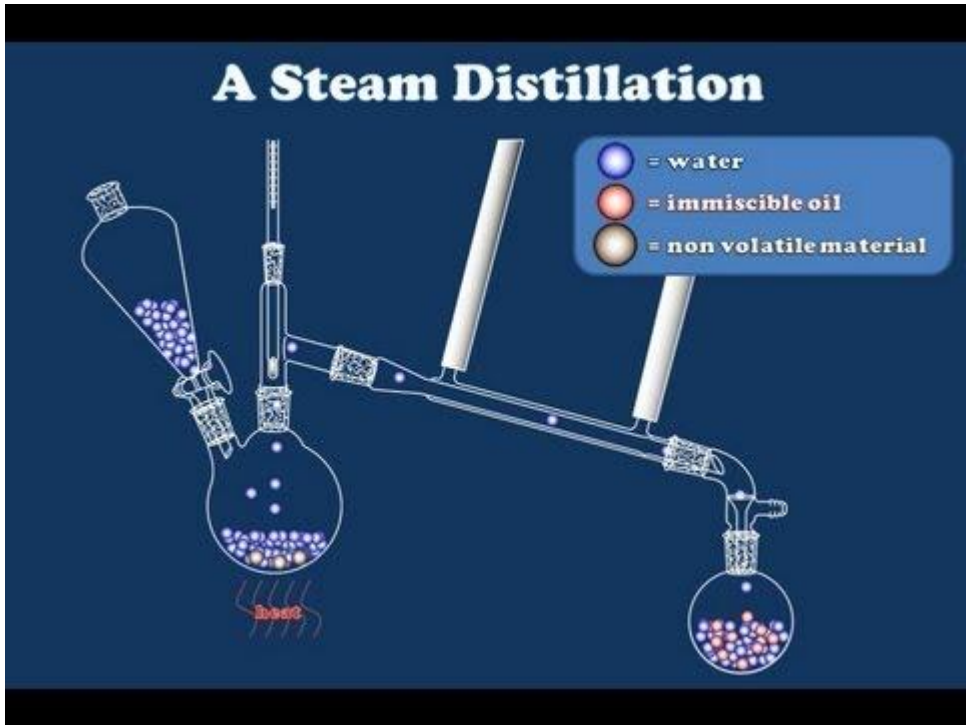
Process

Example

Isolation of Citral

Video of How Steam Distillation Works ?

Steam Distillation



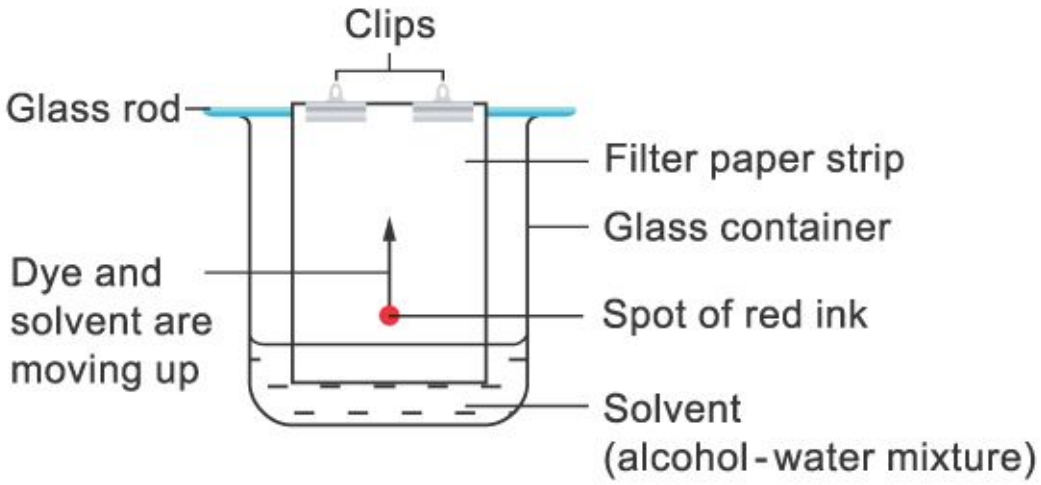
By Using Chromatography

Chromatography

- An effective method of separating a complex mixture of substances
- Paper chromatography is a common type of chromatography

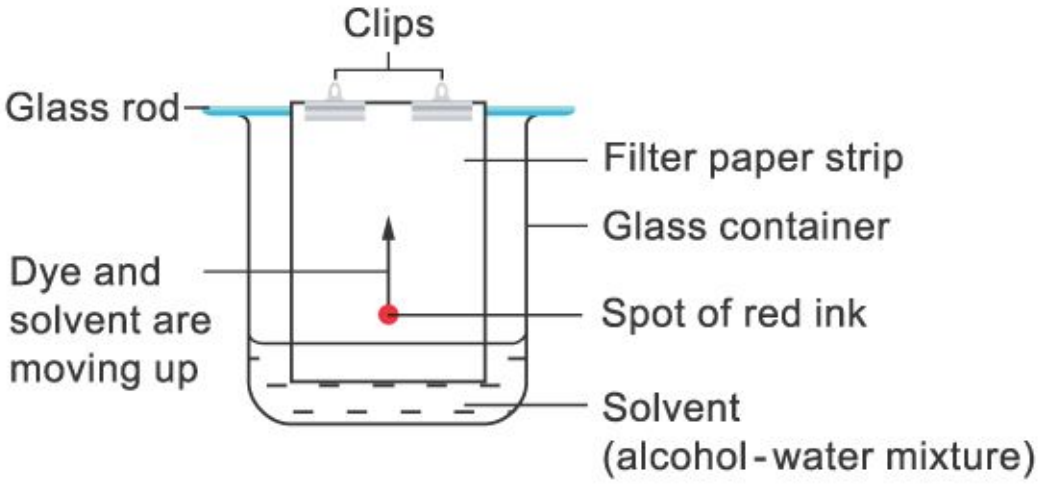
Chromatography

- A solution of the mixture is dropped at one end of the filter paper



Chromatography

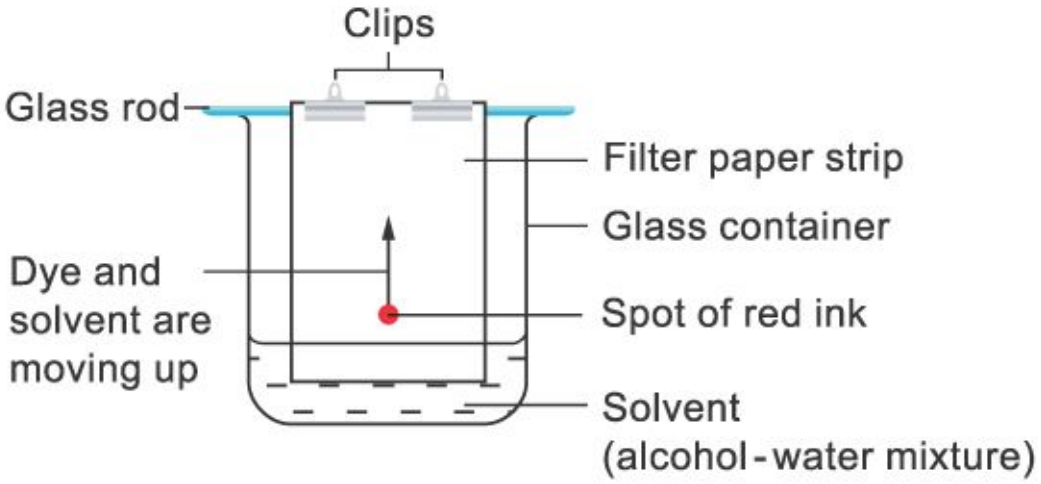
- The thin film of water adhered onto the surface of the filter paper forms the stationary phase
- The solvent is called the mobile phase or eluent



Chromatography

- When the solvent moves across the sample spot of the mixture,

  ➔ partition of the components between the stationary phase and the mobile phase occurs



Chromatography

- As the various components are being adsorbed or partitioned at different rates,

  ➔ they move upwards at different rates

- The ratio of the distance travelled by the substance to the distance travelled by the solvent

  ➔ known as the R_f value

  ➔ a characteristic of the substance

$$k = \frac{C_s}{C_m}$$

- → K is the partition coefficients of the components of a mixture between two immiscible phases - stationary phase and mobile phase
- → C_s is the concentration of the substance in the stationary phase (Adsorbent)
- → C_m is the mobile phase (Eulent)

By Using Sublimation

2. SUBLIMATION

Aim

To separate volatile solids, which pass directly into vapour state on heating from a non-volatile solid.

Principle

A mixture of solid substances, such as camphor, benzoic acid, ammonium chloride, iodine etc., containing non-volatile substances, when heated, change directly into vapour without passing through the liquid state.

Process

Fig :-Sublimation

Substance	Mp	Substance	Mp
1,4-dichlorobenzene	55	Benzoic acid	122
Naphthalene	82	Salicylic acid	159
1-Naphthol	96	Camphor	177
Acetanilide	114	Caffeine	235