# SEPARATION TECHNIQUES - INTRODUCTION

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# DEFINITION:

Separation techniques are those techniques that can be used to separate two different states of matter such as liquids and solids.

Separation processes or a separation method or simply a separation is methodology to attain any mass transfer phenomenon that convert a mixture of substances into two or more distinct product mixtures.

Separation is an important asset to purify component of interest from a mixtures.

# NEED OF SEPARATION TECHNIQUES:

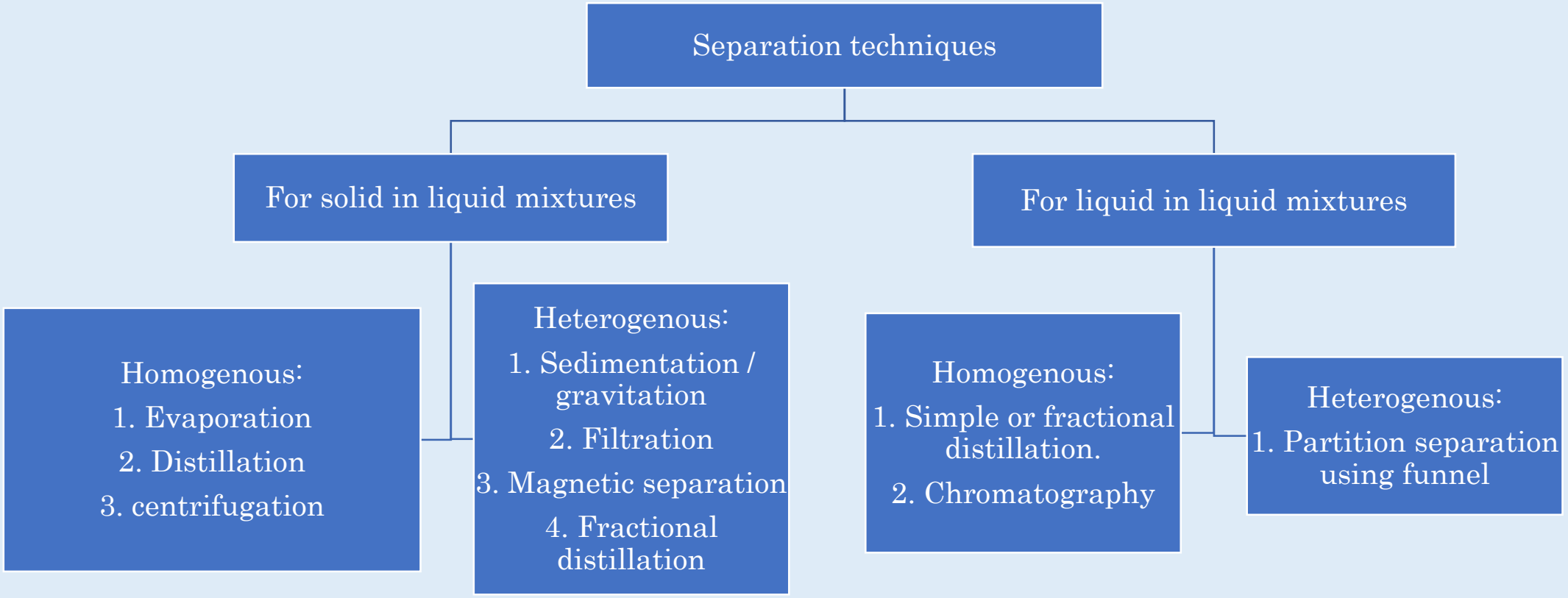
TO IDENTIFY WHAT TO BE SEPARATED FROM MIXTURE.

TO OBTAIN IMPORTANT AND PURE SUBSTANCES.

TO REMOVE UNWANTED PARTICLES.

# TYPES OF SEPARATION TECHNIQUES:

- Separation techniques are classified based on type of mixtures:

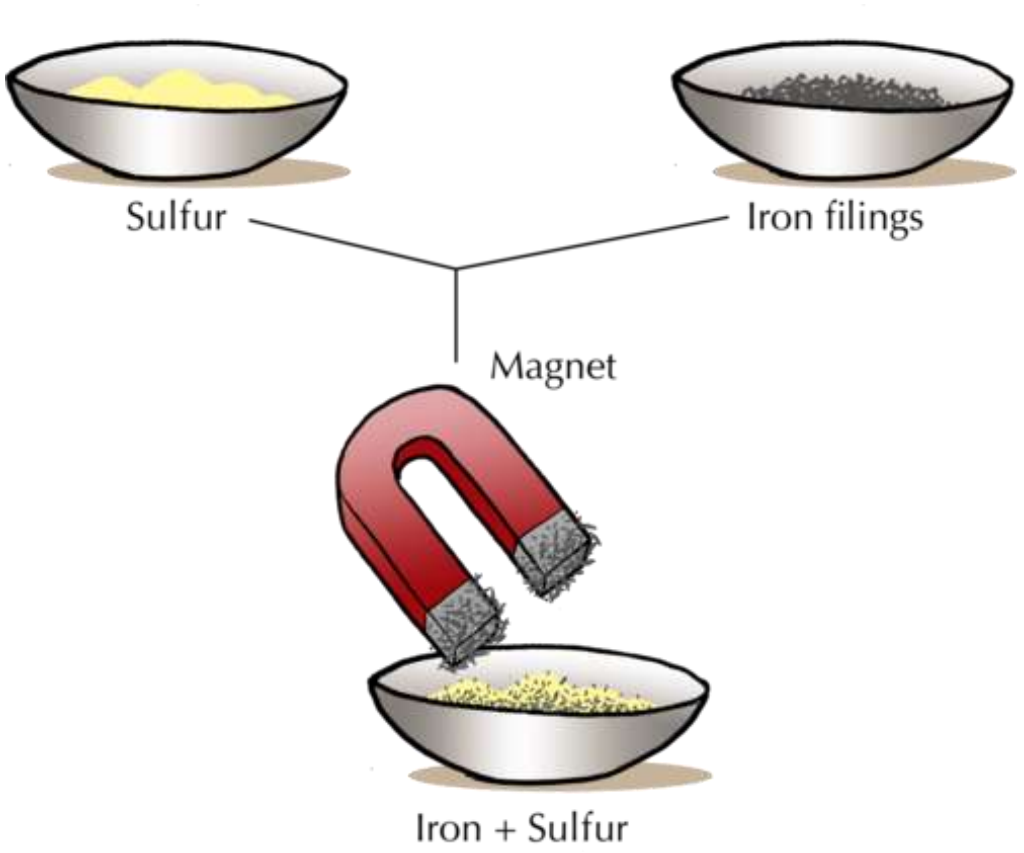


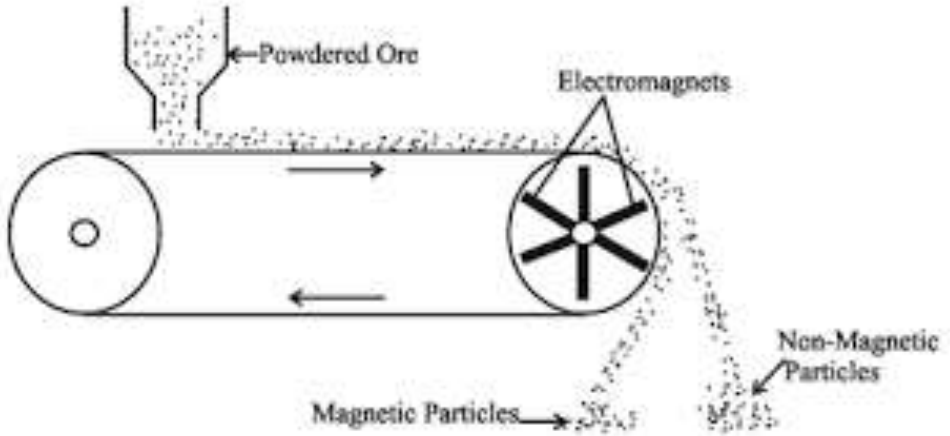
# MAGNETIC SEPARATION:

This method involves the separation of magnetic substances from non-magnetic substances by means of magnet.

Takes advantage of physical property of magnetism, so it useful only for certain substances such as ferromagnetic (materials strongly affected by magnetic fields) and paramagnetic (materials that are less affected, but the effect is still noticeable).

This method involves the separation of magnetic substances from non-magnetic substances by means of magnet.


Sulfur
Iron filings
Magnet
Iron + Sulfur


Powdered Ore
Electromagnets
Non-Magnetic Particles
Magnetic Particles

- Applications:
  - Waste management, low-magnetic field separation in water purification and separation of complex mixtures.
  - To remove metal contaminants from pharmaceutical product streams.
  - Magnetic cell separation. It is currently being used in clinical therapies, more specifically in cancer and hereditary diseases researches.
  - These techniques are combined with PCR (polymerase chain reaction), to increase sensitivity and specificity of results.

# DECANTATION:

**Decantation** is a process for the separation of mixtures of immiscible liquids or of a liquid and a solid mixture such as a suspension.

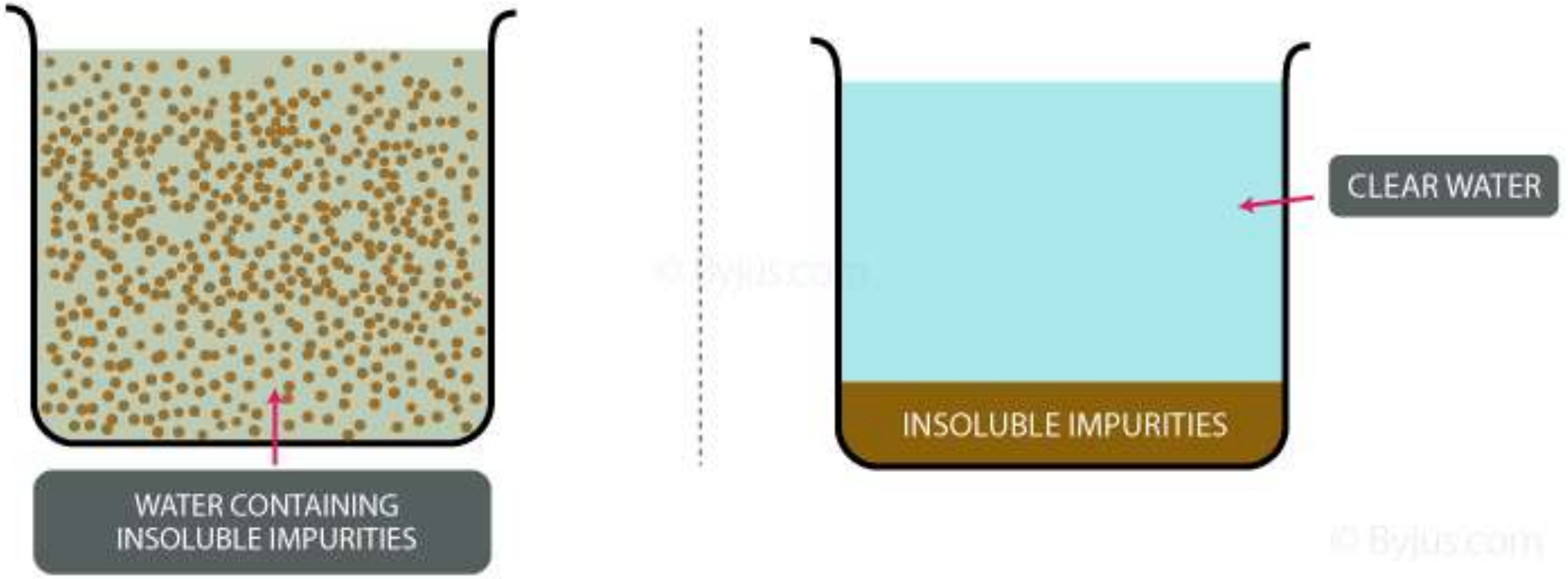
## Immiscible liquid separation:

- Takes advantage of differences in density of the liquids.
- A separatory funnel is an alternative apparatus for separating liquid layers. It has a valve at the bottom to allow draining off the bottom layer. It can give a better separation between the two liquids.
- Example: separation of mixture of oil and water.

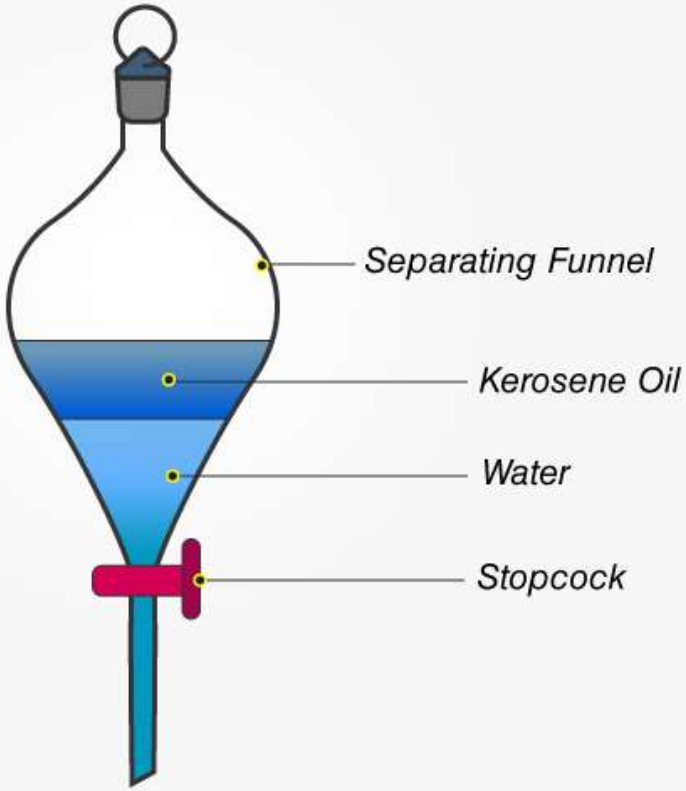
# Liquid-solid separation:

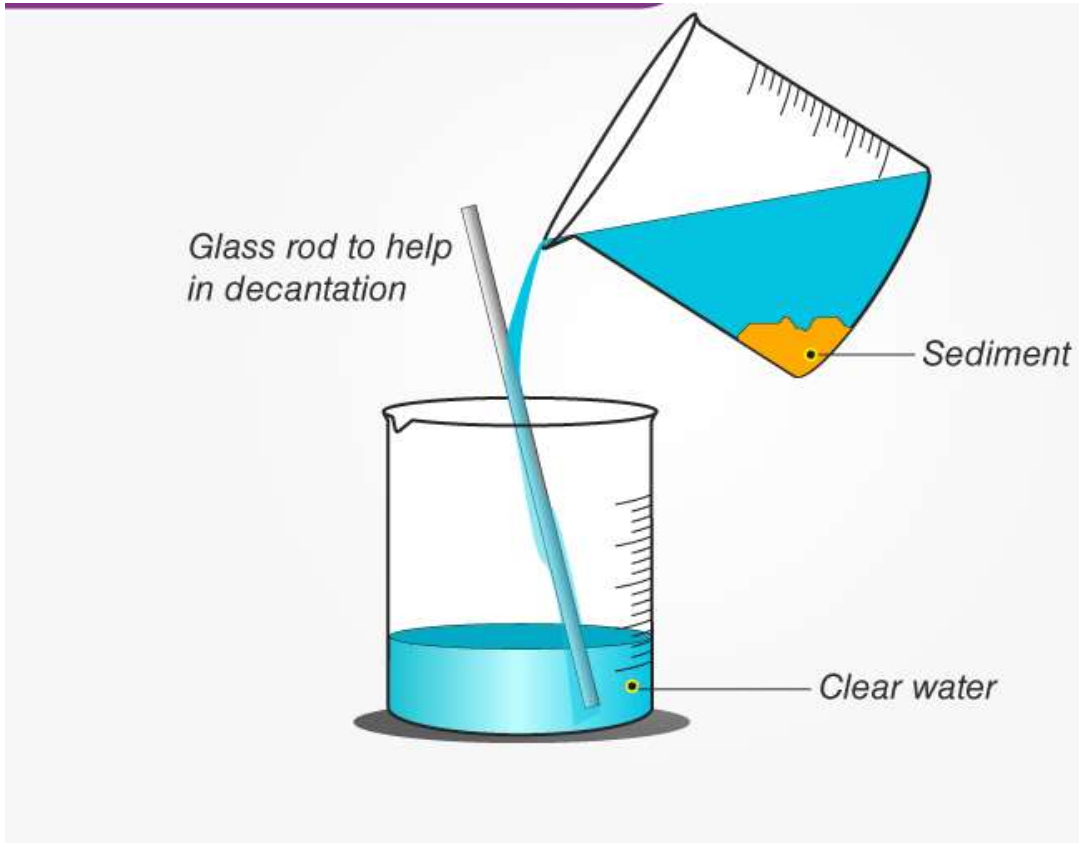
- Takes advantage of gravity/sedimentation of solids in case solid-liquid separation.
- **Sedimentation:** The tendency of particles in suspension to settle down in the fluid due to certain forces like gravity, centrifugal acceleration, or electromagnetism is called as sedimentation.
  - The solid that gets settled down is called as sediment.
- In laboratory it can be done in test tubes. To enhance productivity test tubes should be placed at 45° angle to allow the sediments to settle at the bottom of the apparatus.
- A decanter centrifuge may be used for continuous solid-liquid separation.

# SEDIMENTATION



## DECANTATION



## Examples/applications:

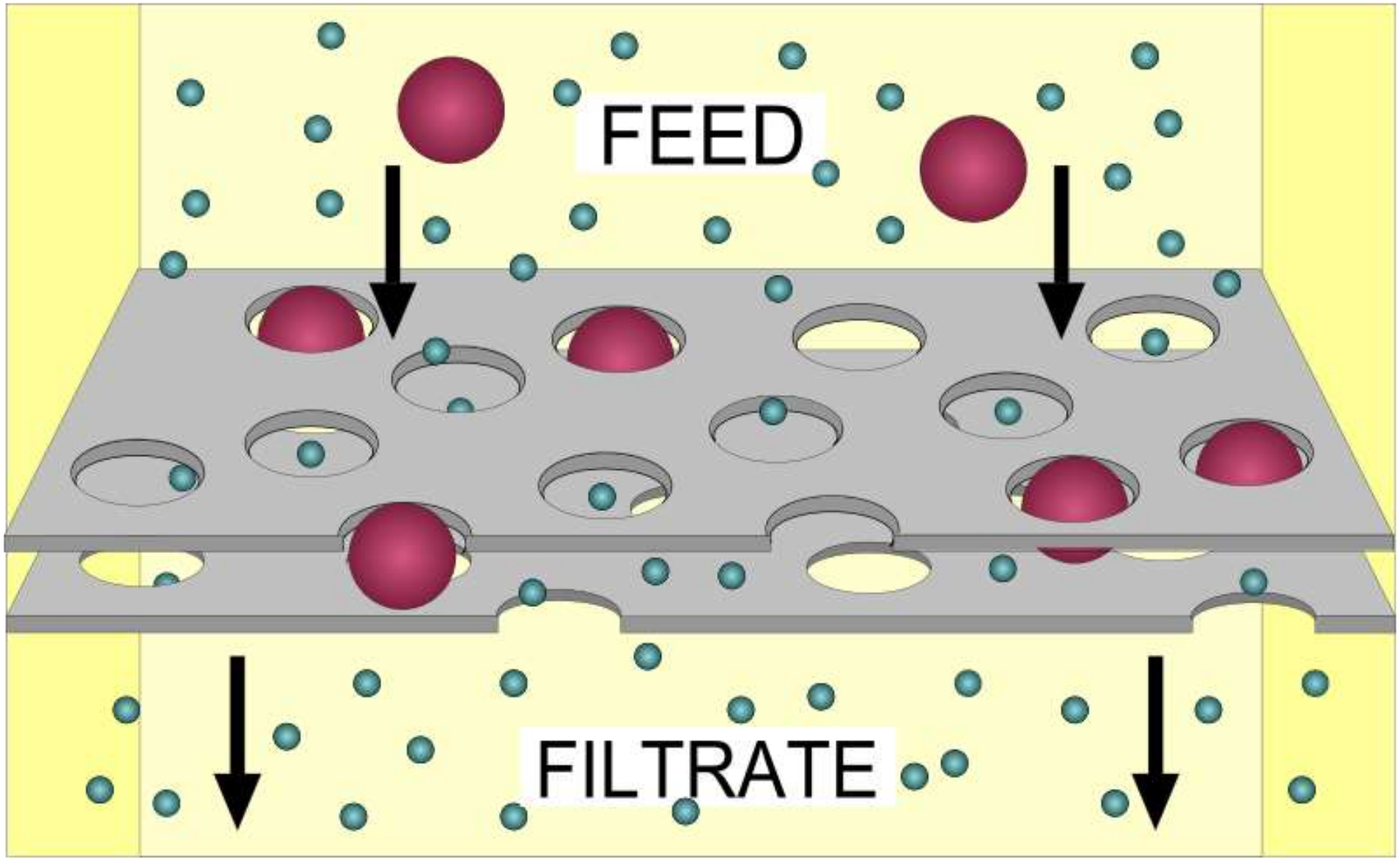
- Decantation is frequently used to purify a liquid by separating it from a suspension of insoluble particles.
- Decantation is also present in nanotechnology. In the synthesis of high-quality silver nanowire (AgNW) solutions and fabrication process of high-performance electrodes, decantation is also being applied which greatly simplifies the purification process.
- Fat is determined in butter by decantation.
- In sugar industry, processing of sugar beets into granular sugar many liquid - solid separations are encountered.

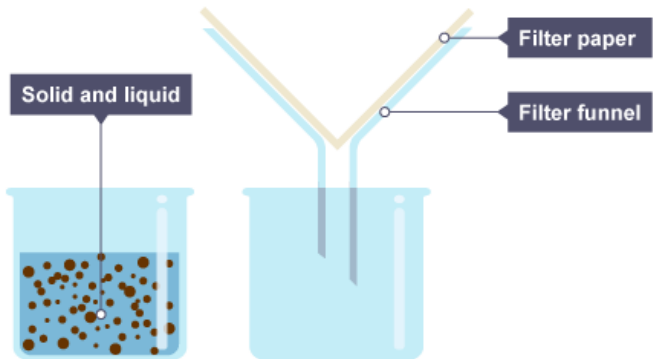
**FILTRATION:**

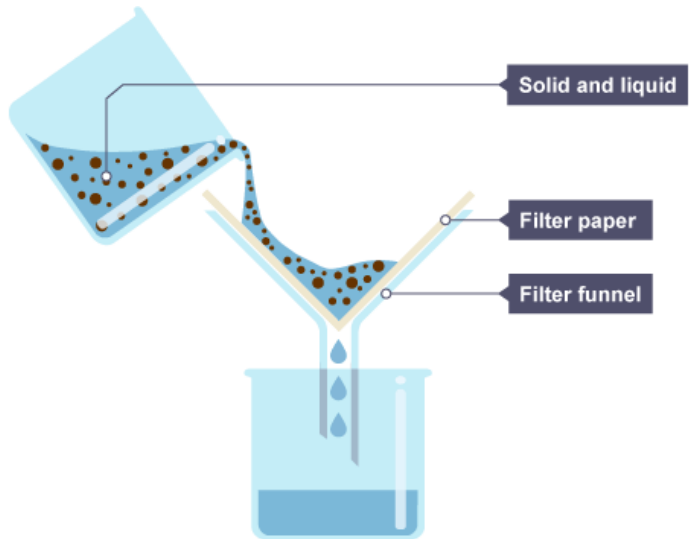
Takes advantage of physical property of state of matter, its size and solubility in liquid.

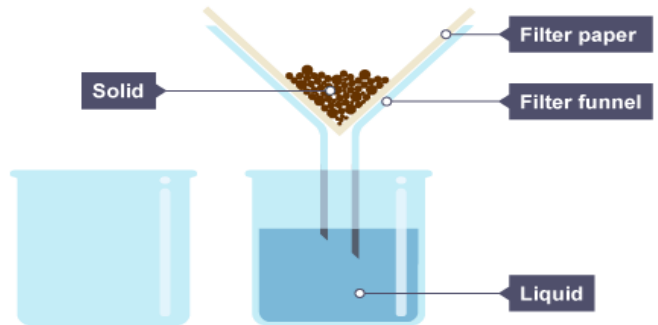
Mechanical, physical or biological operations that separates solids from fluids (liquids or gases ) by adding a medium through which only the fluid can pass can be called as filtration.

- The substance which is left behind in the filtration medium is called **residue**.
- The liquid which passes through the filtration medium is called **filtrate**.


FEED
FILTRATE


Solid and liquid
Filter paper
Filter funnel


Solid and liquid
Filter paper
Filter funnel


Solid
Filter paper
Filter funnel
Liquid

# Applications:

- Most important techniques used by chemists to purify compounds.
- HEPA filters in air conditioning to remove particles from air.
- In the laboratory, a Büchner funnel is often used, with a filter paper serving as the porous barrier.

# CENTRIFUGATION:

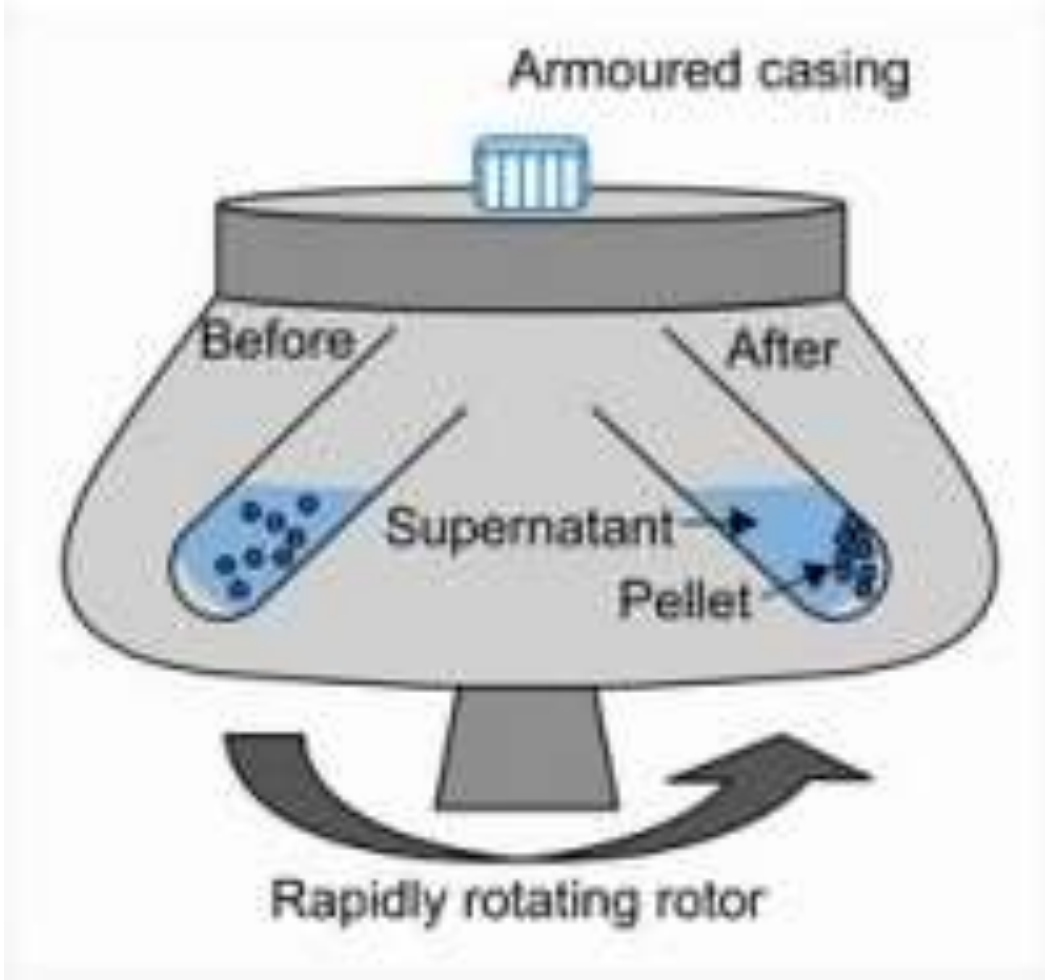
It is used to separate mixtures where the solid particles don't settle faster and which are of very small size and can't be separated by filtration.

**Centrifugation** is a technique which involves the application of centrifugal force to separate particles from a solution according to their size, shape, density, viscosity of the medium and rotor speed.

A **centrifuge** is a device that separates particles from a solution through use of a rotor.

More-dense components of the mixture migrate away from the axis of the centrifuge (move to the outside), while less-dense components of the mixture migrate towards the axis, i. e., move to the center.

The rate of centrifugation is specified by the angular velocity usually expressed as revolutions per minute (RPM), or acceleration expressed as *g*. The conversion factor between RPM and *g* depends on the radius of the centrifuge rotor.


Armoured casing
Before
After
Supernatant
Pellet
Rapidly rotating rotor

# Applications/examples:

- Microcentrifuges are used to process small volumes of biological molecules, cells, or nuclei.
- Used in diagnostic laboratories for blood and urine tests.
- Aids in separation of proteins using purification techniques such as salting out.
- Differential Centrifugation used to separate organelles and membranes found in cells.

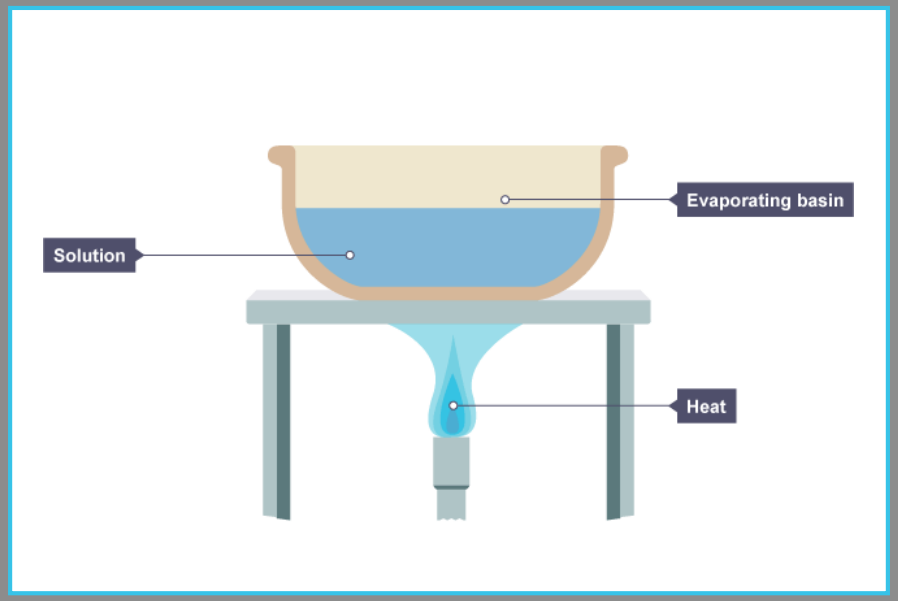
# EVAPORATION:

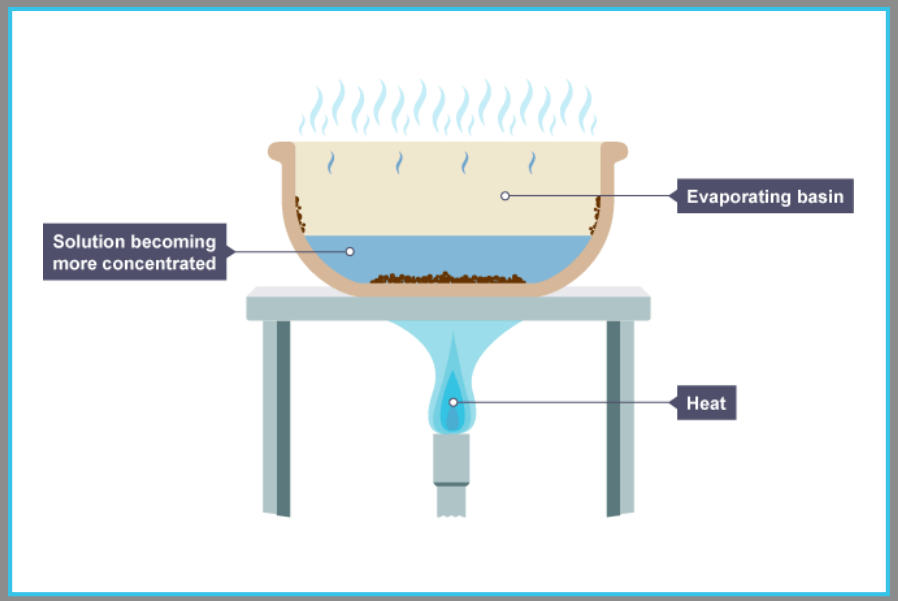
Evaporation is type of vaporization that occurs on the surface of liquid as it changes into gas phase.

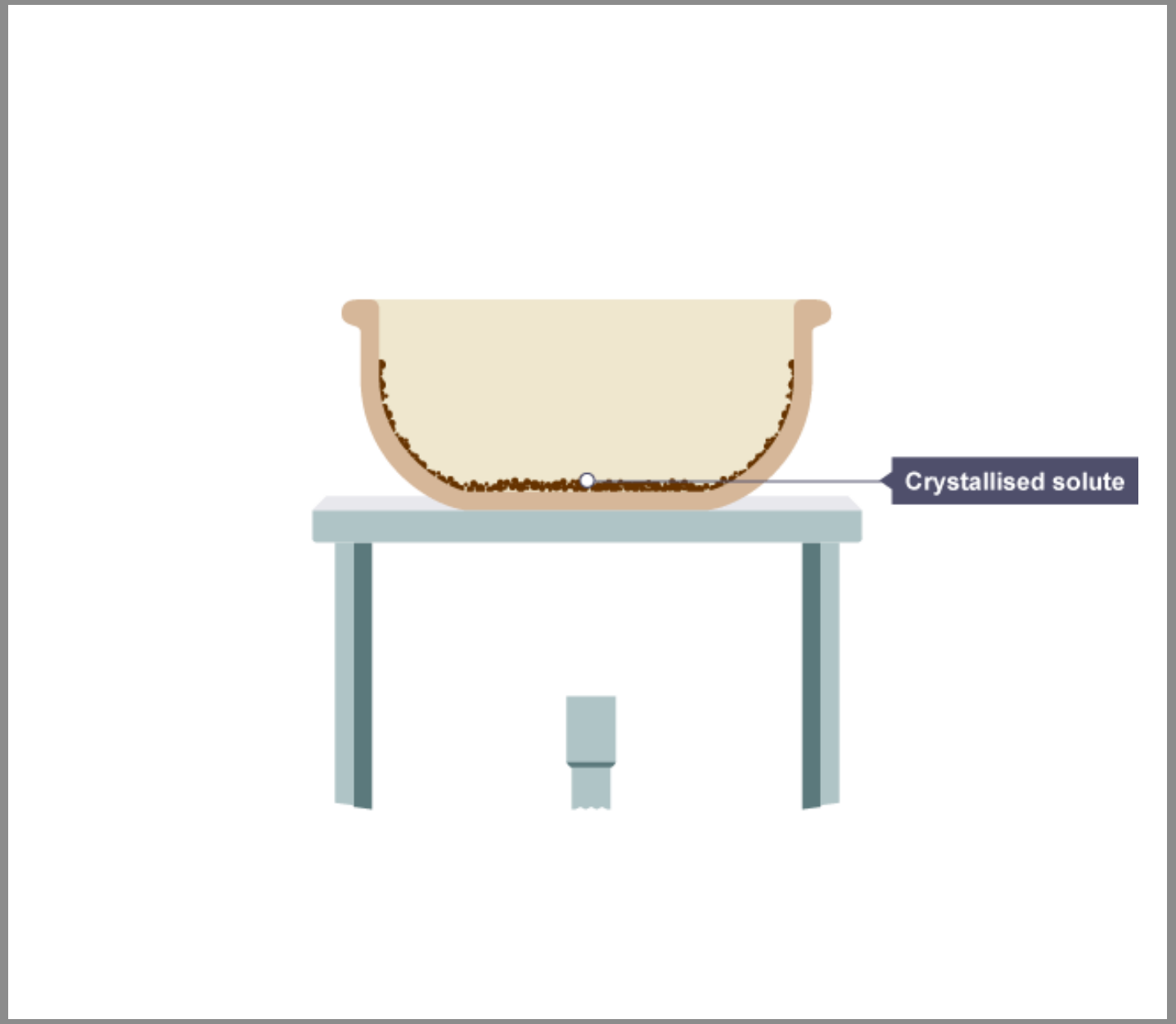
This separation technique can be used to separate solutes that are dissolved in solvent, by boiling the solution. The solvent gets vaporized leaving back the solute.

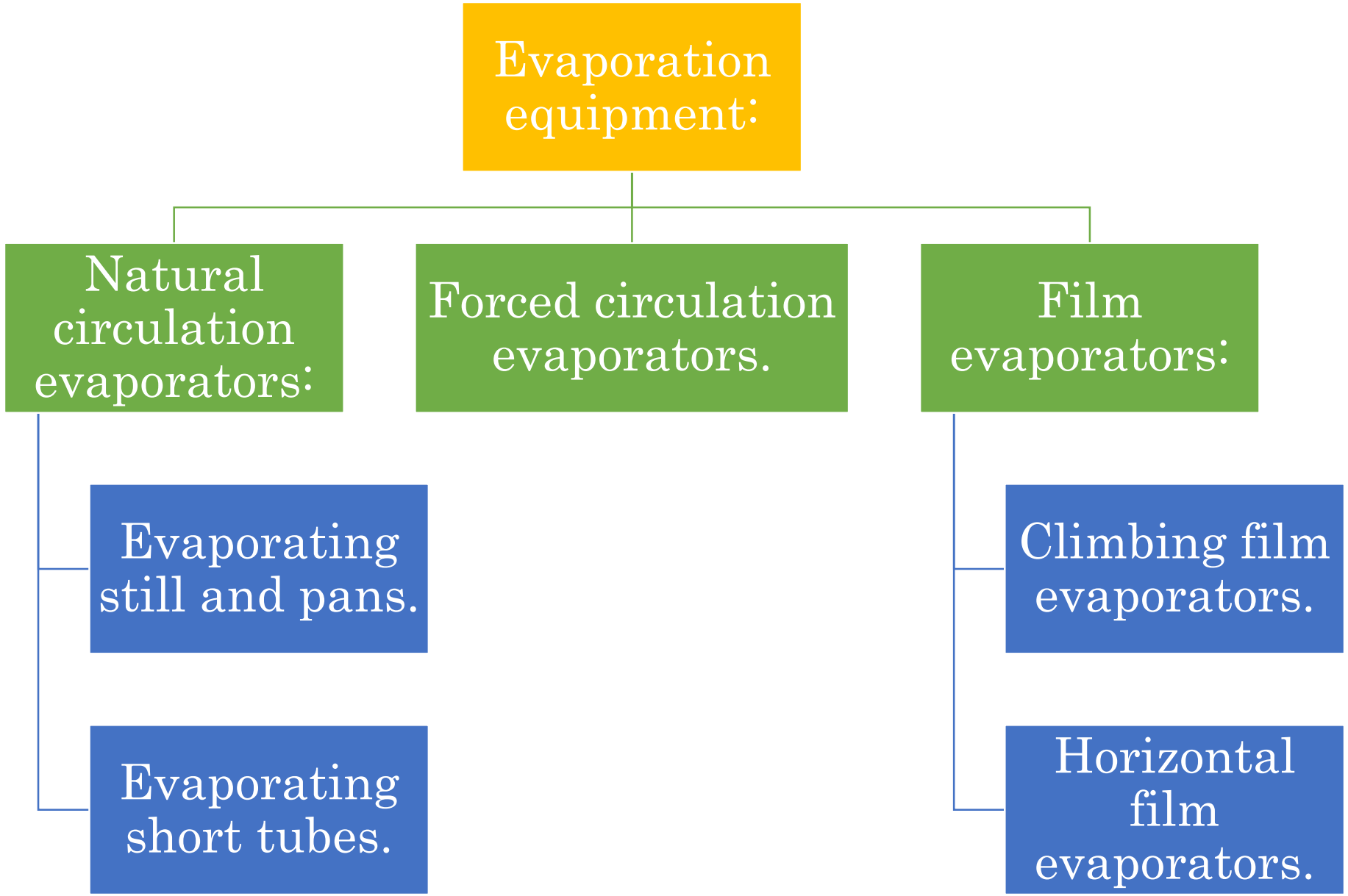
Factors affecting:

- Concentration of the substance evaporating in the air.
- Flow rate of air, pressure, temperature.
- Surface area.
- Intermolecular forces.


Evaporating basin
Solution
Heat


Evaporating basin
Solution becoming more concentrated
Heat


Crystallised solute


Evaporation equipment:
Natural circulation evaporators:
Forced circulation evaporators.
Film evaporators:
Evaporating still and pans.
Evaporating short tubes.
Climbing film evaporators.
Horizontal film evaporators.

# Applications/examples:

- Recovering salts from solution.
- The use of evaporation to dry or concentrate samples is a common preparatory step for many laboratory analyses such as spectroscopy and chromatography.
- Demineralization of water.

# DISTILLATION:

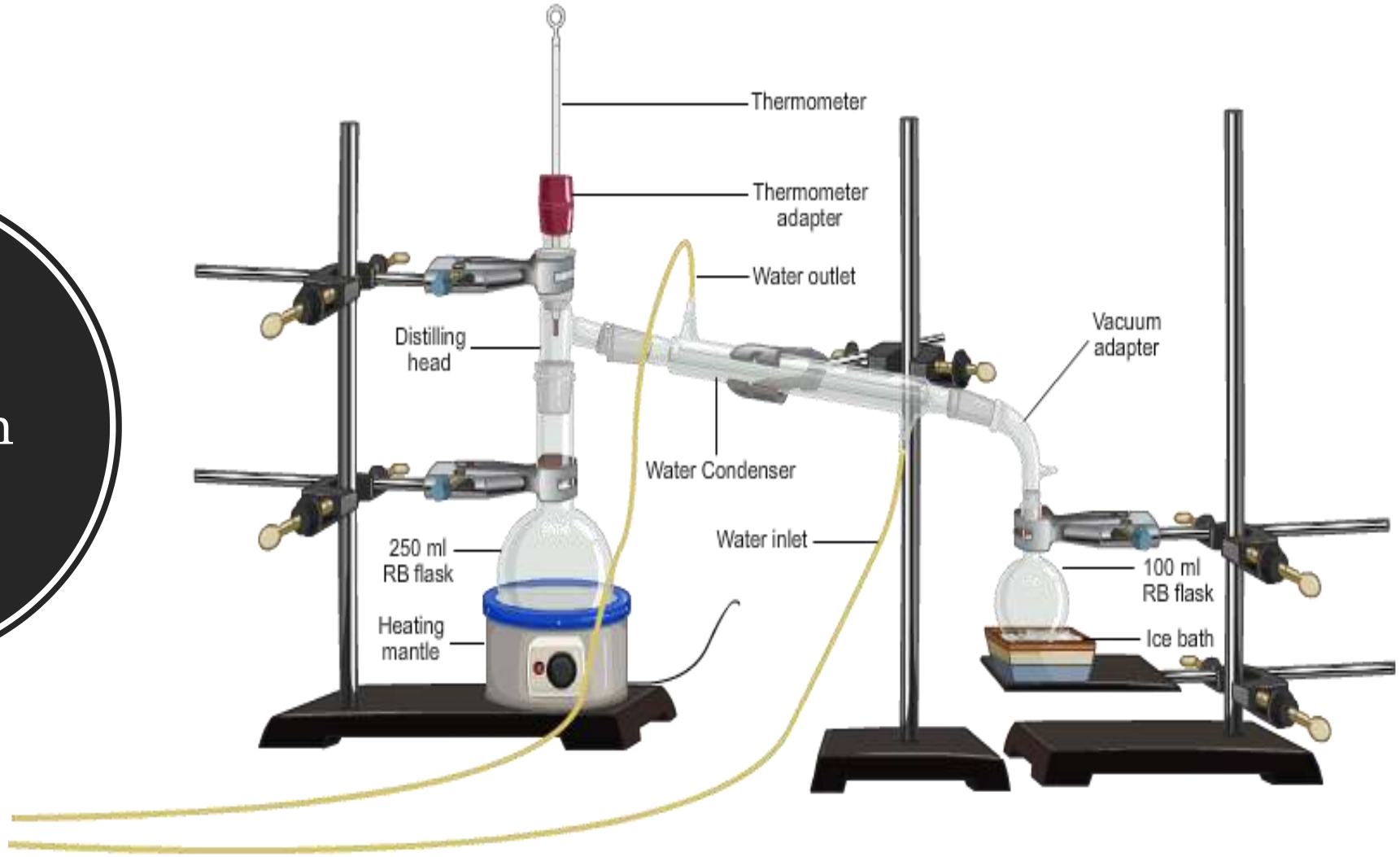
**Distillation** is an effective method to separate mixtures comprised of two or more pure liquids (called “components”).

The separation of a mixture of liquids based on the physical property of **boiling point**.

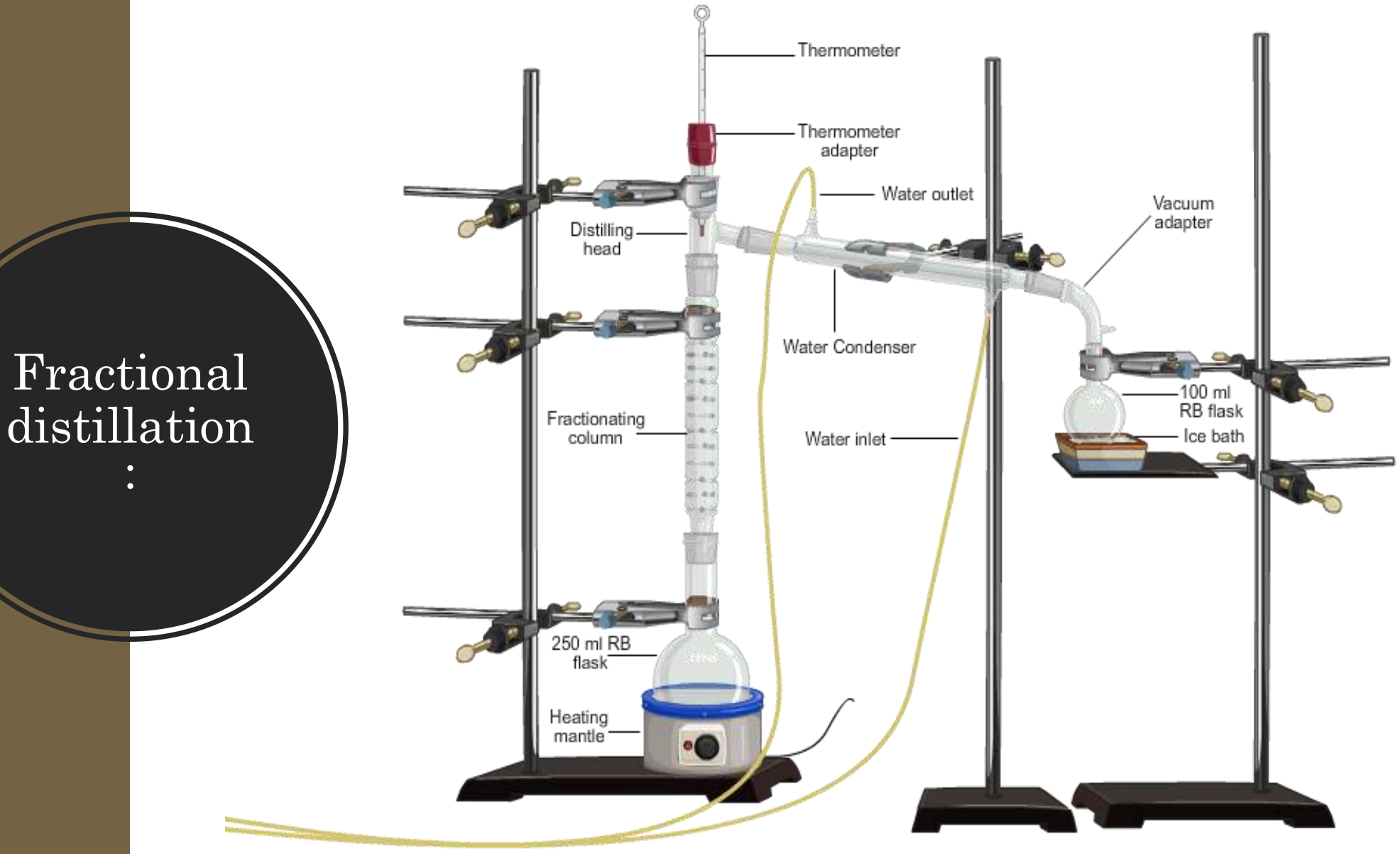
Distillation is a purification process where the components of a liquid mixture are vaporized and then condensed and isolated.

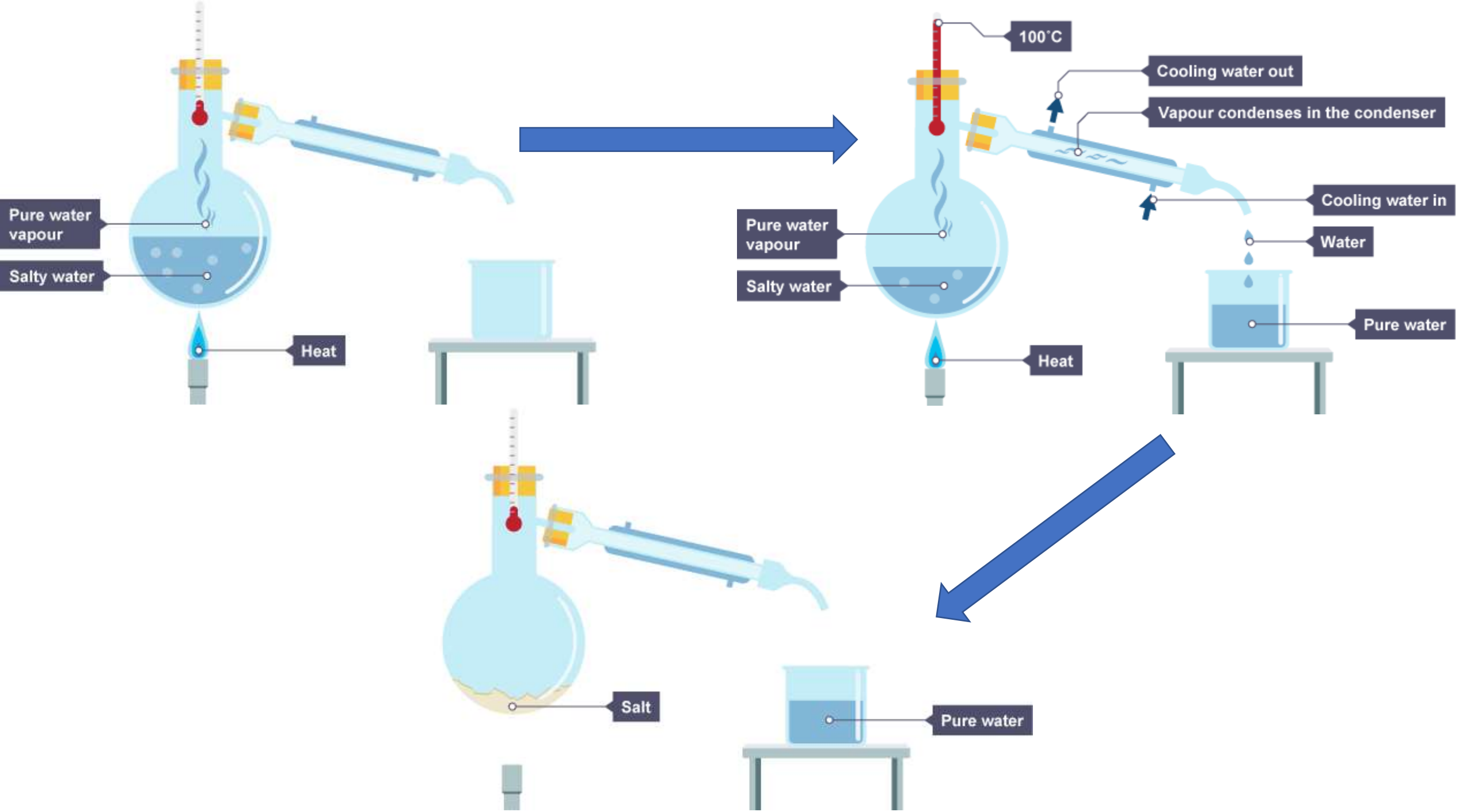
The mixture is heated until one of the components boils (turns to a vapor). The vapor is then fed into a condenser, which cools the vapor and changes it back into a liquid that is called ***distillate***. What remains in the original container is called the "**residue**".

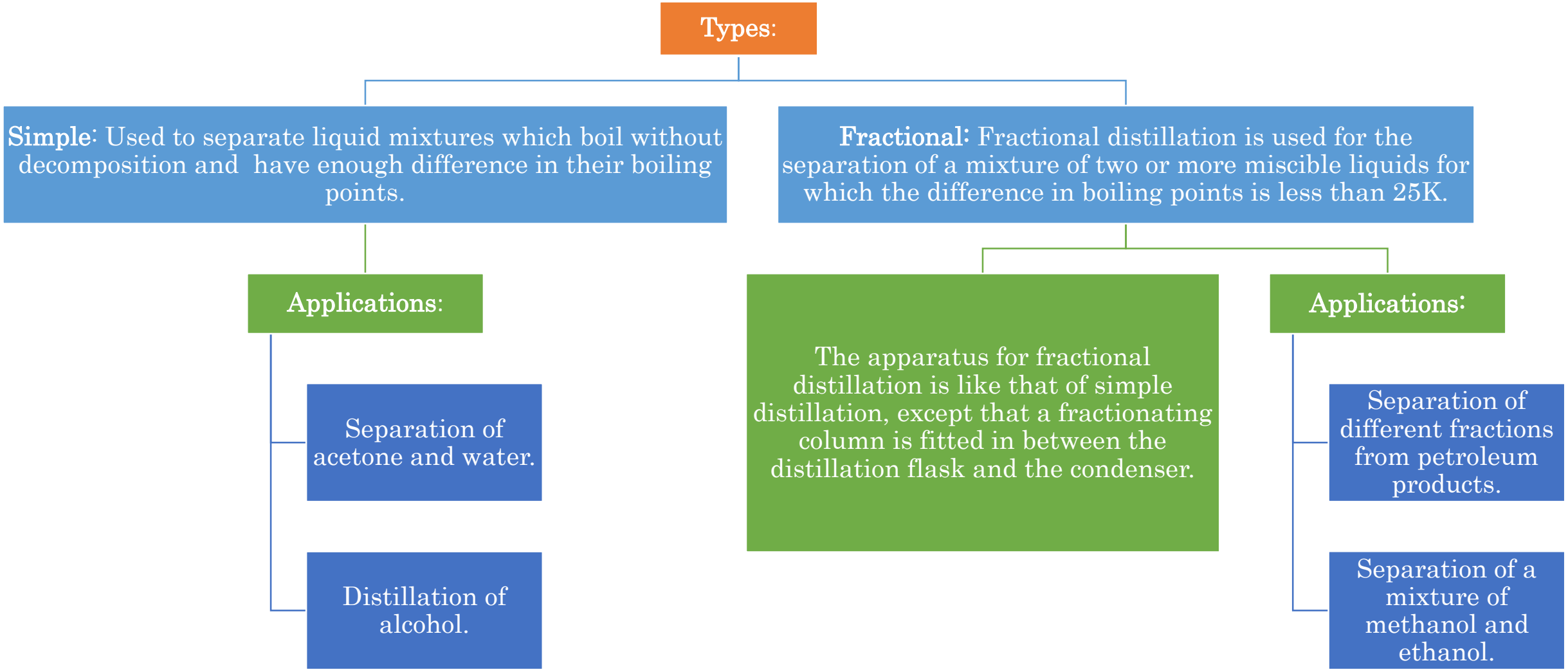
# Simple distillation :



# Fractional distillation :




Pure water vapour
Salty water
Heat
100°C
Cooling water out
Vapour condenses in the condenser
Cooling water in
Water
Pure water
Pure water vapour
Salty water
Heat
Salt
Pure water


Types:
Simple: Used to separate liquid mixtures which boil without decomposition and have enough difference in their boiling points.
Fractional: Fractional distillation is used for the separation of a mixture of two or more miscible liquids for which the difference in boiling points is less than 25K.
Applications:
Separation of acetone and water.
Distillation of alcohol.
The apparatus for fractional distillation is like that of simple distillation, except that a fractionating column is fitted in between the distillation flask and the condenser.
Applications:
Separation of different fractions from petroleum products.
Separation of a mixture of methanol and ethanol.

# Other applications/examples:

- Separation of volatile oils.
- Separation of drugs obtained from plant or animal sources – vitamin A from fish liver oil.
- Purification of organic solvents.
- Manufacture of official preparations – spirit of nitrous ether, spirit of ammonia, distilled water, and water for injection.
- Refining petroleum products.

# CRYSTALLIZATION:

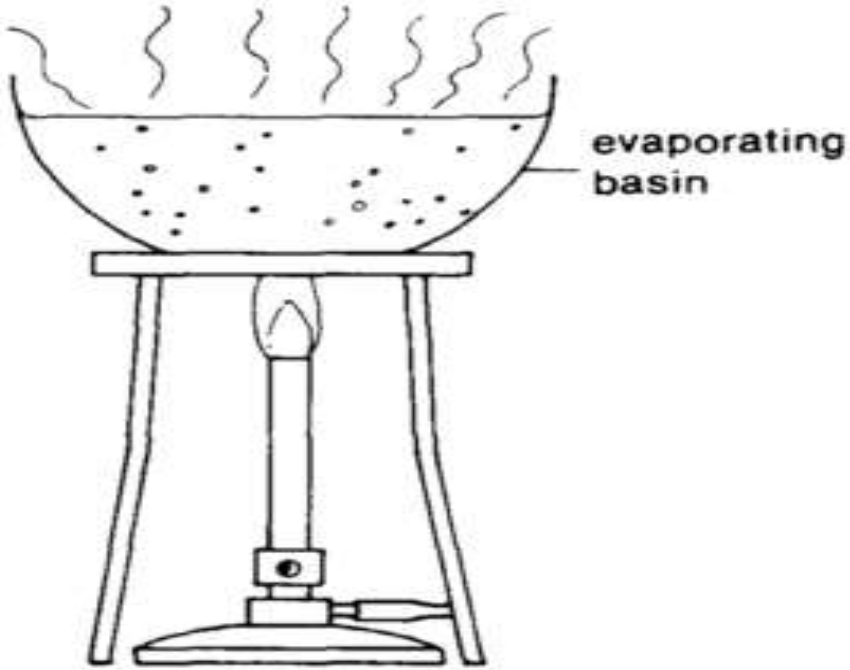
- **Crystallization** is the (natural or artificial) process by which a solid forms, where the atoms or molecules are highly organized into a structure known as a crystal.
- Used to separate a dissolved heat-liable (will decompose upon heating and hence can sublime) solid (solute) from a solution.
- Most minerals and organic molecules crystallize easily, and the resulting crystals are generally of good quality, i.e. without visible defects.

First, the solution is heated to evaporate off most of the solvent to make a hot and nearly saturated solution.

After which, the hot solution to is kept for cooling, naturally. The solubility of the solute decreases as the solution is cooled, and the excess solute which can no longer be dissolved in the saturated solution crystallizes out of the solution.

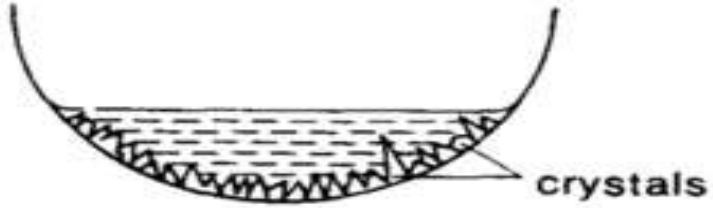
The crystals which are formed can be separated from the remaining solution by filtration.

**Step 1:**



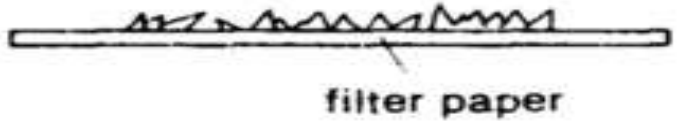

The solution is heated to evaporate most of the solvent.

**Step 2:**




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

**Step 3:**



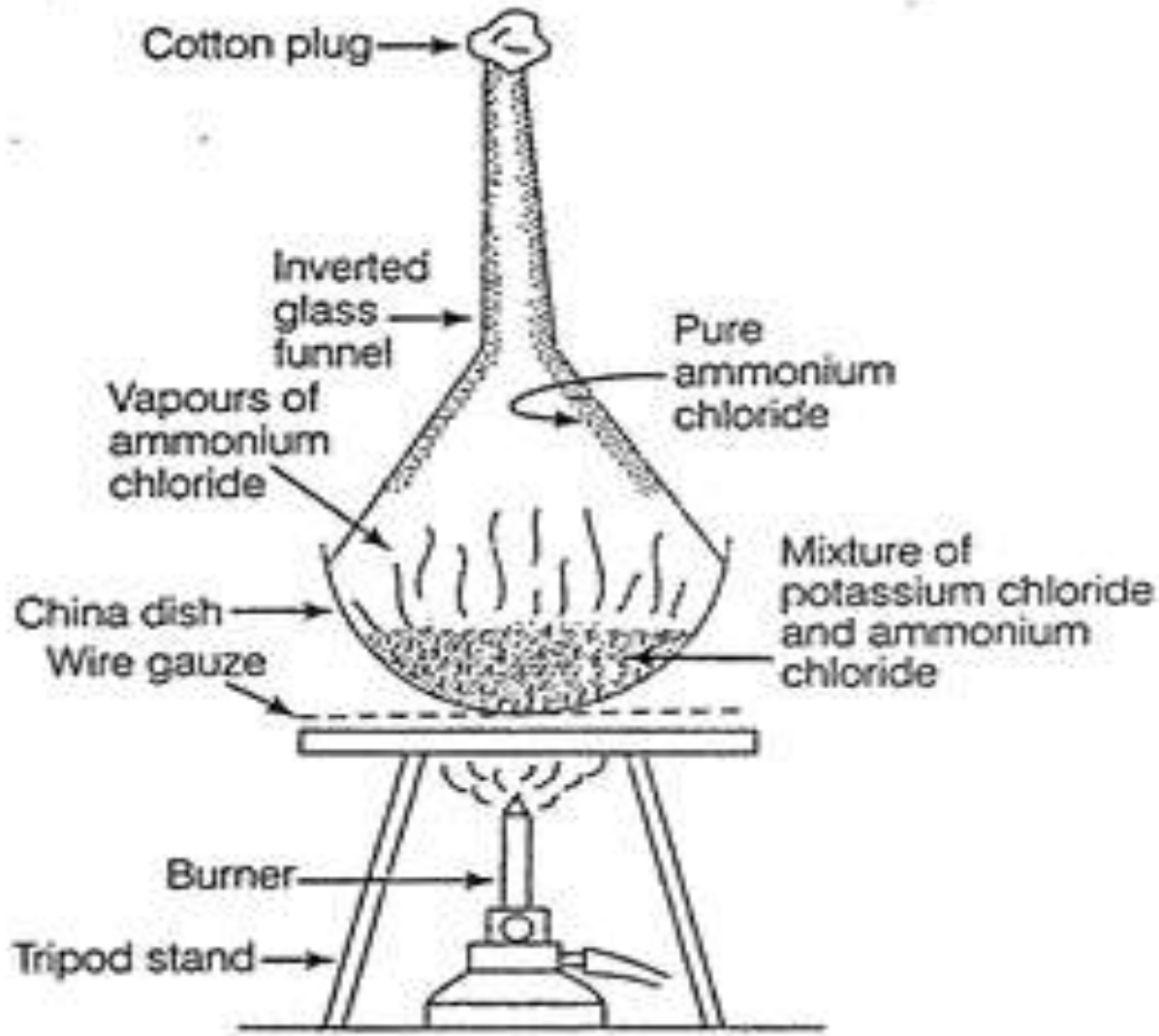

The cold solution is poured off to obtain the crystals. The crystals may be dried by pressing them between sheets of filter paper.

Applications/ examples:

- Purification of drugs.
- Separation of crystals of alum from impurities sample.
- **Fractional Crystallization** : It is possible to separate mixtures of different ionic compounds having **identical chemical composition** by dissolving them in water and adjusting the temperature of the solution so that one compound crystallizes out and the other does not.
  - E.g. $K_2SO_4$ and $KNO_3$

# SUBLIMATION SEPARATION:

- **Sublimation** is the transition of a substance directly from the solid to the gas phase, without passing through the intermediate liquid phase.
- This technique takes the advantage of substance's sublimable property.
- Separate a mixture of solids containing one which sublimes and one (or more than one) which does not, by heating the mixture.

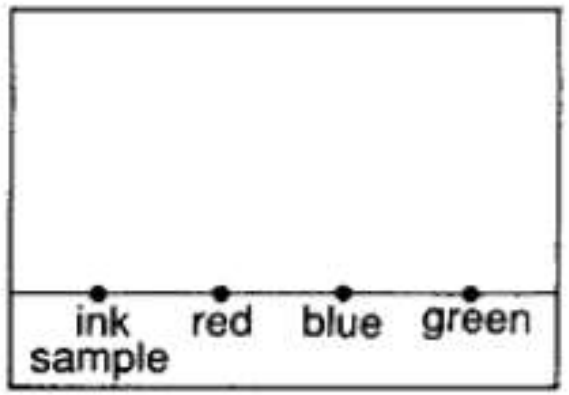

Cotton plug
Inverted glass funnel
Pure ammonium chloride
Vapours of ammonium chloride
Mixture of potassium chloride and ammonium chloride
China dish
Wire gauze
Burner
Tripod stand

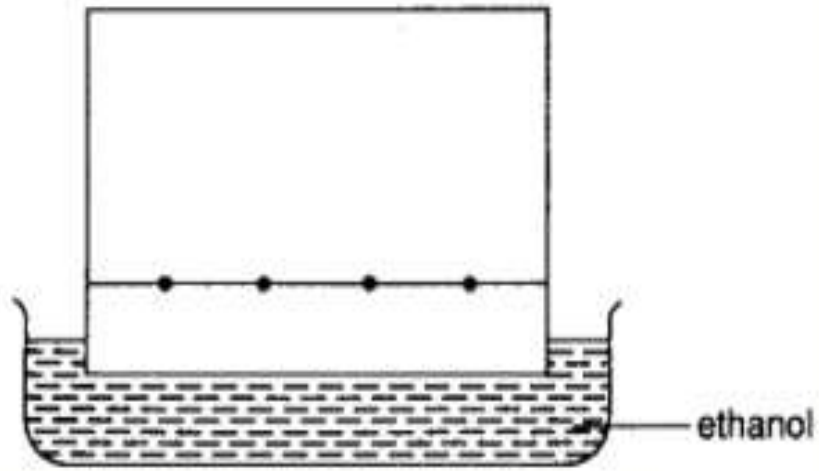
# Applications/examples:

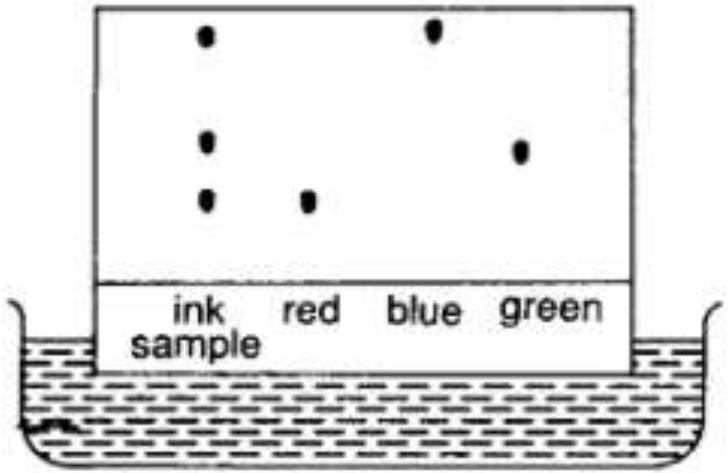
- Separate iodine from sand.
- Dye-sublimation printers help in rendering digital pictures in a detailed and realistic fashion which helps in the analysis of substances.
- Sublimation finds practical application in forensic sciences.
- Chemists usually prefer sublimation as a purification method to purify volatile compounds.

# CHROMATOGRAPHY:

- Chromatography is a separation technique used to separate the different components in a liquid mixture.
- The mixture is dissolved in a fluid called the *mobile phase,* which carries it through a structure holding another material called the *stationary phase.*
- Chromatography is vast separation technique which has many methods or has different principles of separation involved.


ink
sample
red
blue
green


ethanol


ink
sample
red
blue
green

| Type of Chromatography | Applications in the Real World | Why and What is it |
|---|---|---|
| **Liquid Chromatography** | testing water samples to look for pollution | Used to analyze metal ions and organic compounds in solutions. It uses liquids which may incorporate hydrophilic, insoluble molecules. |
| **Gas Chromatography** | detecting bombs in airports, identifying and quantifying such drugs as alcohol, being used in forensics to compare fibres found on a victim | Used to analyze volatile gases. Helium is used to move the gaseous mixture through a column of absorbent material. |
| **Thin-Layer Chromatography** | detecting pesticide or insecticide residues in food, also used in forensics to analyze the dye composition of fibres | Uses an absorbent material on flat glass plates. This is a simple and rapid method to check the purity of the organic compound. |
| **Paper Chromatography** | separating amino acids and anions, RNA fingerprinting, separating and testing histamines, antibiotics | The most common type of chromatography. The paper is the stationary phase. This uses capillary action to pull the solutes up through the paper and separate the solutes. |

# EXTRACTION TECHNIQUES:

- Involves the separation of medicinally active portions of plant or animal tissues from the inactive or inert components by using selective solvents in standard extraction procedures.
- Different methods of extraction are:

| Method | Solvent | Temperature | Pressure | Time | Volume of organic solvent consumed | Polarity of natural products extracted |
|---|---|---|---|---|---|---|
| Maceration | Water, aqueous and non-aqueous solvents | Room temperature | Atmospheric | Long | Large | Dependent on extracting solvent |
| Percolation | Water, aqueous and non-aqueous solvents | Room temperature, occasionally under heat | Atmospheric | Long | Large | Dependent on extracting solvent |
| Decoction | Water | Under heat | Atmospheric | Moderate | None | Polar compounds |
| Reflux extraction | Aqueous and non-aqueous solvents | Under heat | Atmospheric | Moderate | Moderate | Dependent on extracting solvent |
| Soxhlet extraction | Organic solvents | Under heat | Atmospheric | Long | Moderate | Dependent on extracting solvent |
| Pressurized liquid extraction | Water, aqueous and non-aqueous solvents | Under heat | High | Short | Small | Dependent on extracting solvent |
| Supercritical fluid extraction | Supercritical fluid (usually S-$CO_2$), sometimes with modifier | Near room temperature | High | Short | None or small | Nonpolar to moderate polar compounds |
| Ultrasound assisted extraction | Water, aqueous and non-aqueous solvents | Room temperature, or under heat | Atmospheric | Short | Moderate | Dependent on extracting solvent |
| Microwave assisted extraction | Water, aqueous and non-aqueous solvents | Room temperature | Atmospheric | Short | None or moderate | Dependent on extracting solvent |
| Pulsed electric field extraction | Water, aqueous and non-aqueous solvents | Room temperature, or under heat | Atmospheric | Short | Moderate | Dependent on extracting solvent |
| Enzyme assisted extraction | Water, aqueous and non-aqueous solvents | Room temperature, or heated after enzyme treatment | Atmospheric | Moderate | Moderate | Dependent on extracting solvent |
| Hydro distillation and steam distillation | Water | Under heat | Atmospheric | Long | None | Essential oil (usually non-polar) |

# THANK YOU