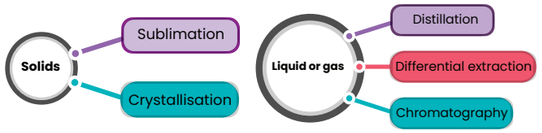
Purification of Organic Compounds

Methods of purification



Sublimation

- Solid is converted directly into vapour without undergoing liquid state.

eg: Purification of iodine, Camphor, Naphthalene, ammonium chloride etc.

Crystallisation:

- It is based on the difference in the solubilities of the compound & the impurities in a suitable solvent.
- The impure compound is dissolved in a solvent in which it is sparingly soluble at room temperature but appreciably soluble at higher temperature.
- If the compound is highly soluble in one solvent and very little soluble in another solvent, crystallisation can be satisfactorily carried out in a mixture of these solvents.
- eq: Purification of sugar, purification of potash alum etc.

Distillation :

Principle : Based on difference in b.p. of components of mixture.

- Also used to separate : Volatile liquids from nonvolatile impurities

Types of distillation:

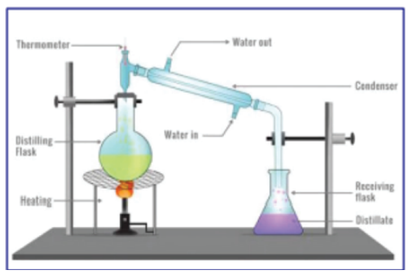
Simple distillation :
To separate liquids which have sufficient difference in b.p
eg: chloroform (334K) & aniline (457K)

Fractional distillation:
To separate liquids which have small difference in b.p.
eg: To separate different fractions of crude oil in petroleum industry.

Vacuum distillation
(distillation under reduced pressure)
This method is used to purify liquids having very high boiling points but they decompose at or below their boiling points.
eq: Glycerol is separated from spent - lye in soap industry.

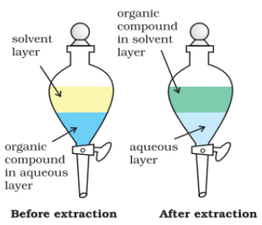
Steam Distillation
- To separate substances which are steam volatile and are immiscible with water.

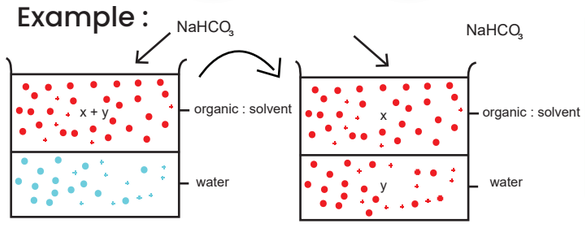
eg: Aniline, Nitrobenzene, o- nitro phenol.

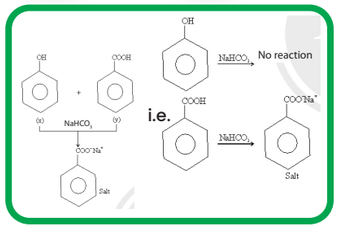


Differential Extraction Principle:-

- Based on difference in solubility of organic compounds in different solvents.
- When an organic compound is present in an aqueous medium, it is separated by shaking it with an organic solvent in which it is more soluble than in water.
- The organic solvent & aq: solvent should be immiscible with each other so that they form 2 distinct layers which can be separated by separatory funnel.
- The org. solvent is later removed by distillation or evaporation to get back org. comp.







Q. A mixture of camphor and benzoic acid can be separated by:
(a) Sublimation
(b) Extraction with a solvent
(c) Chemical method
(d) Fractional distillation

Q. The best method for the separation of naphthalene and benzoic acid from their mixture is:
(a) Chromatography
(b) Crystallisation
(c) Distillation
(d) Solvent extraction

Q. In steam distillation, the sum of the vapour pressure of the volatile compound and that of water is:
(a) Equal to atmospheric pressure
(b) Less than atmospheric pressure
(c) More than atmospheric pressure
(d) Exactly half of the atmospheric pressure

Q. A liquid compound can be purified by steam distillation only if it is
(b) Steam volatile, miscible with water
(c) Not steam volatile, immiscible with water
(d) Steam volatile, immiscible with water

Chromatography

PHYSICS WALLAH

Latest technique

To purify compound — **Chromatography is used** — To separate mix

To test purity of compound

Stationary phase — Solid/liq.

In Chromatography there are 2 phases

Mobile phase — Pure solvent or mix. of solvents or gases

Types of chromatography

1) Adsorption Chromatography:

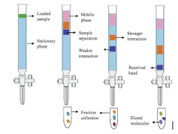
- Based on the fact that different components of a mixture have different degrees of adsorption on adsorbent (silica gel or alumina)

Stationary phase - solid
Moblie phase - liquid/gas

2 Types
- Column Chromatography
- Thin layer Chromatography (TLC)

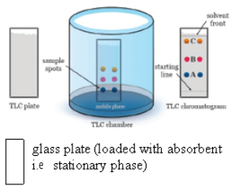
a) Column Chromatography

- Involves separation of a mixture over a column of adsorbent (stationary phase) packed in a glass tube.
- Component of the mixture which is more soluble in stationary phase is adsorbed first than the component which is less soluble in stationary phase.
- The most readily adsorbed substances are retained near the top and others come down to various distances in the column



b) Thin layer chromatography (TLC)

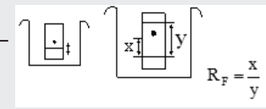
Involves separation of substances of a mixture over thin layer of an adsorbent coated on glass plate.
The components of the mixture move up along with the eluent to different distances depending on their degree of adsorption and separation takes place.



Retardation factor:-

express degree of adsorption of each Component of mixture.

$$R_f = \frac{\text{distance moved by component of mixture from base line}}{\text{distance moved by solvent (mobile phase) from base line.}}$$

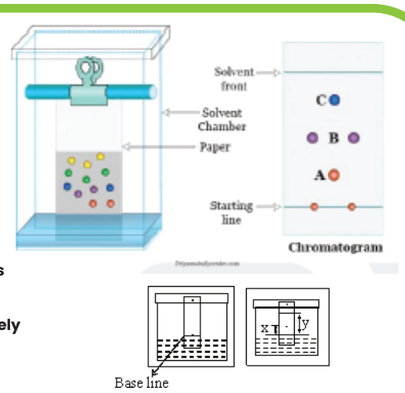


2) Partition chromatography:

- Based on continuous differential partitioning of components of a mixture between stationary & mobile phase. Also known as **liquid paper chromatography**

Stationary phase - liquid
Mobile phase – liquid

- **A special quality paper known as chromatography paper is used.**
eg: cellulose paper
- **Chromatography paper contains water trapped in it, which works as Stationary phase.**
- **Moving phase is a solvent or a mixture of solvents in which spotted chromatography paper is suspended.**
- **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 paper strip so developed is called Chromatogram**



Q. Paper chromatography is an example of :
(a) Partition chromatography
(b) Thin layer chromatography
(c) Column chromatography
(d) Adsorption chromatography

Q. The most suitable method of separation of 1 : 1 mixture of ortho & para-nitrophenols is
(a) Steam distillation
(b) Sublimation
(c) Chromatography
(d) Crystallisation