METHODS OF PURIFICATION OF SOLIDS

- **Crystallisation**: A saturated solution of impure substance is prepared in hot solvent and is heated with vegetable or animal charcoal which adsorbs the impurities. The solution is filtered while hot through a hot water funnel. The filtrate on cooling deposits crystals of pure compound. Success of the process depends upon the selection of the solvent. The impurities must be least soluble while hot and most-soluble while cold. The quick cooling gives tiny but pure crystals while slow cooling gives bigger but impure crystals. When crystal formation is initiated by adding crystals of substance it is called "**Seeding**".
- **Fractional crystallisation** : It is based on the differential solubilities of different compounds in a solvent. The compound having less solubility crystallises out first on cooling leaving behind others in solution. Sometimes mixture of two solvents eg. Alcohol & water; Chloroform & Petroleum ether, Alcohol or ether give better results.
- **Sublimation**: Some solids directly pass into vapour when heated and vapour directly pass into solid when cooled without the intermediate formation of liquid. This is known as sublimation. The substances which sublime can be purified by this method provided the impurities present do not sublime. Camphor, naphthalene, anthracene, benzoic acid, phthalic anhydride and anthraquinone are purified by sublimation.

METHODS OF PURIFICATION OF LIQUIDS

- **Simple distillation**: The vaporisation of a liquid by heating and subsequent condensation of vapours by cooling is known as distillation. The liquids boiling under ordinary conditions of temperature and pressure without decomposition and containing non volatile impurities are purified by simple distillation.
- **Fractional distillation**: It is employed for separating two or more volatile liquids having boiling points close to each other eg. acetone (bpt 60°C) and methanol (bpt 65°C). The vapours of the liquids pass through the fractionating column which provides greater space for their cooling. The vapours of high boiling fractions condense and fall back into distillation flask. The process is repeated till fractions of high volatility go up followed by of lower volatility. They are collected separately.
- **Distillation under reduced pressure or vacuum distillation**: Some liquids decompose when heated to their boiling points eg. glycerol. Such liquids can be purified by distillation under reduced pressure much below their boiling points. The lower the pressure lower is the boiling point and vice versa.
- **Steam distillation**: The liquids insoluble in water, steam volatile in nature, having high molecular weight and having high vapour pressure are purified by steam distillation provided the impurities present are not steam volatile.

  Theory of steam distillation: The liquid boils when its vapour pressure becomes equal to the atmospheric pressure. For a mixture of liquid (pl) and steam (ps) we have at boiling point P = pl + ps pl = (P – ps)
  Hence liquid will boil at lower temperature than its normal boiling point. Examples Aniline, o-nitrophenol, bromobenzene, salicylaldehyde, essential oils etc.

CHROMATOGRAPHIC METHODS

It was discovered by Tswett (1906) and developed by Kuhn, Winterstein and Karrer.

PRINCIPLE

Selective adsorption or distribution of various components of mixture between the two phases - fixed phase and mobile phase.

CLASSIFICATION

- **Adsorption chromatography**: Fixed phase - Solid or ion exchange resin

  Mobile phase - Liquid or gas
  Hence it is further known as liquid - solid chromatography, gas-solid chromatography or ion exchange chromatography.
- **Partition Chromatography**: Fixed phase - liquid supported on inert solid

  Mobile phase - liquid or gas
  Hence we have liquid - liquid partition chromatography and liquid gas partition chromatography
  Paper chromatography is the example of partition chromatography

COLUMN CHROMATOGRAPHY
Example of Adsorption chromatography
Adsorbents used are: alumina, silica gel, cellulose powder, sucrose, animal charcoal, magnesium oxide or kieselguhr etc.
Liquid Solvents used are : benzene, petroleum ether, chloroform, alcohol etc.
When the solvent is poured over the mixture present at the top of a column packed with adsorbent the components are separated into number of layers called **Zones**, **bands** or **chromatograms** due to preferential adsorption. The process being known as development.
Elution : The continuous pouring of solvent from the top of column is known as elution or running of column.
Solvent : It is known as **eluent**.
The most weakly adsorbed component is eluted first by least polar solvent while more strongly adsorbed component is eluted later by highly polar solvents.

CHEMICAL METHODS

The substance to be purified is treated with a suitable chemical reagent to form a stable derivative (impurities being unreacted). It is then separated by suitable method and decomposed to get the pure compound. Examples:

- Mixture of amines (1°, 2° and 3°) is separated by Hinsberg's method and Hoffmann's method.
- Acetic acid from pyroligneous acid is separated by forming Ca salt.
- Acids are separated by forming Na derivatives with $NaHCO_3$.
- Commercial benzene contains thiophene which is removed by forming sulphonic acid derivative.
- Carbonyl compounds are purified by forming bisulphite derivative.
- Absolute alcohol is obtained from rectified spirit by quicklime process and azeotropic distillation.

EXTRACTION

The process of removing a substance from its aqueous solution by shaking with a suitable solvent is known as extraction. (partition law) The greater the number of operations the greater is the recovery of substance. Soxhlet extractor is used for continuous extraction.