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Chapter-2

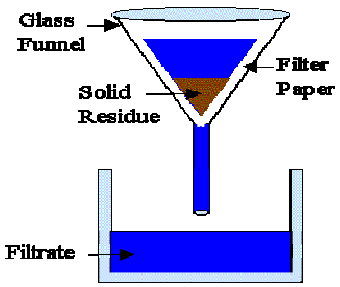
Separations techniques: Methods used in chemistry to purify substances or to isolate them from other substances are called separations techniques.

Examples: Filtration, crystallizations, sublimation, solvent extraction, chromatography etc

Filtration: The process of separating an insoluble solid from a liquid by passing the mixture through a filter paper or porous medium is called filtration.

Basic Principle of Filtration: The basic principle of filtration is that the liquid component of the mixture passes freely from porous medium while the solid component cannot pass through the pores because of bigger size and trapped on the surface of the porous medium.

- Solids which do not dissolve in liquid can be separated by filtration
- filtration is commonly used to separate solid from liquid from mixture of two
- The solid remained in the filter paper is called the residue
- The liquid obtained after passing through the filter paper is called the filtrate
- The level of the liquid should be lower than the brim of filter paper



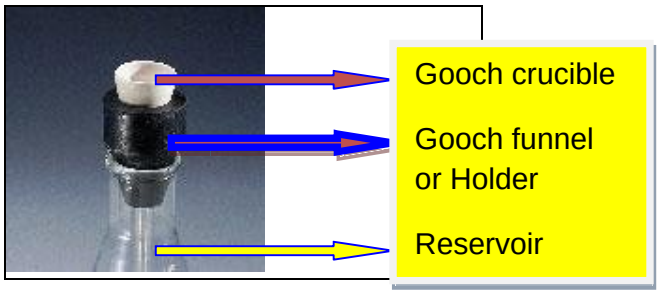
Gooch crucibles: A small crucible with a perforated bottom in which precipitates can be collected,dried, and weighed is called Gooch crucibles or Gooch filter

- Developed by American chemist F. A. Gooch
- It has got numerous small holes at bottom

Procedure for Gooch crucibles:

1. weight the Gooch crucibles

2. Fit it into holders called Gooch funnel
3. Fit filter paper for effective filtration
4. Fit the Gooch funnel into reservoir flask
5. Pass the mixture and dry the precipitate in Gooch crucible
6. Weight the Gooch crucible again with dried precipitate
7. Minus the actual weight of Gooch crucible and you will get the weight of precipitate



Filtration by using Gooch crucible

Sintered glass crucibles: These are crucibles which have got perforated bottom like Gooch crucibles but they do not require filter paper.

- They are made of glass which have got resistance to the most chemicals
- They are readily cleaned
- They can be heated to a temperature up to 400 ^{0}C

Crystallization: The formation of crystal from liquid or gas is called crystallization.

Basic Principle of crystallization: The basic principle of crystallization is that solute is dissolved in suitable solvent at high temperature and when the solution is cooled the pure crystals of the solute are obtained.

- it is used for separation and purification of substances

Sublimations: The process in which solids directly convert to gaseous state without passing from liquid state is called sublimation.

Basic principle of sublimation: The intermolecular forces in some solids are very weak so when surface molecules gain energy it overcome on intermolecular forces and change to vapours directly.

Example NH_4Cl , Naphthalene, benzoic acid etc

- The process of sublimation is used to separate volatile from non volatile solids.

Solvent extraction: The process in which solvent is used to recover the required components from the mixture of solids, liquids or gas is called solvent extraction.

Basic Principle of solvent extraction: The basic principle of solvent extraction is that mixture is contacted with suitable solvent which will dissolve the solutes of interest because different solutes have different solubility in different solvent.

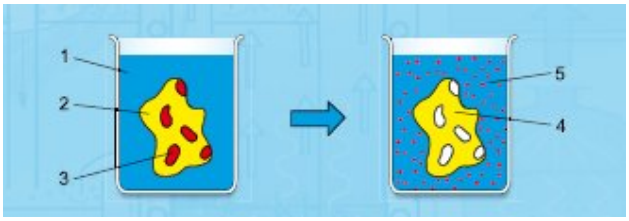
Example: solvent extraction is used to refine petroleum, vegetable oil, chemicals etc

Modes of solvent extraction: Solvent extraction is employed in two modes

1) Solid liquid extraction: The extraction in which required component is removed from solid by liquid solvent is called solid liquid extraction.

Example:

- Metal salts are obtained from ore by solid liquid extraction.
- extraction of oils from pressed oil seed with benzene
- Making tea is also solid liquid extraction because the required component (flavour, caffeine) is dissolved in hot water while tea ground remained.

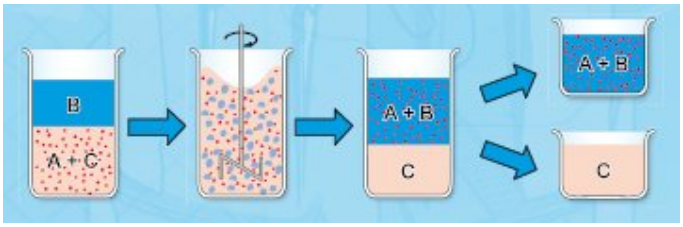


2) liquid-liquid extraction: The extraction in which required component is removed from liquid by liquid solvent is called liquid-liquid extraction. The required components are more soluble in liquid solvent than in carrier liquid.

Example: a. Removal of vitamins from liquid solution

b. Let suppose we have mixture of two liquid A and C and we want to extract A so when suitable solvent B is added to initial solution (A+C). If the component A is more

soluble in B and C is not soluble in it then A will dissolve in B and separate from C. After settlement two phases will be obtained: the extract (A+B) and carrier C



Law of distribution: It is stated as 'a solute distribute itself between two immiscible liquids in a constant ratio of concentration irrespective of the amount of solute added'

Example: When the known amount of iodine is added to known volume of water and tetra chloromethane (CCl_4) mixture. The iodine will distribute itself between the two layers of liquid (aqueous and CCl_4). It is found that the ratio of iodine concentration in both layers remains the same due to dynamic equilibrium and gives constant value called distribution coefficient (k)

$$k = \frac{[I_2 \text{ in water}]}{[I_2 \text{ in } CCl_4]} = 1.17 \times 10^{-2}$$

The value of k remains constant at constant temperature and has no unit. From above method iodine can be extracted from aqueous solution by CCl_4.

Chromatography: The technique which is used for separation of complex mixture into its respective components is called chromatography.

In chromatography substances of complicated mixture are separated on the basis of their polarity. Chromatographic systems are always constructed with two main phases

1) Stationary phase: The phase which is immobilized during the flow of mixture component is called stationary phase. It may be solid or liquid.

   Example: silica gel, aluminium oxide, cellulose paper etc

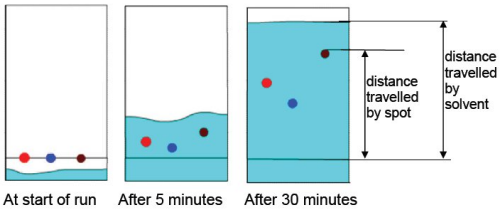
2) Mobile phase: The phase which is mobilized and shifts the components of mixture against stationary phase is called mobile phase.

   Example: Various organic solvents

Procedure for paper chromatography.

1. The sample is dissolved in an appropriate solvent to make about one percent solution
2. The samples are spotted with the help of capillary glass onto the paper
3. The sample spots are placed at certain distance from each other

4. The spots are first dried and then paper is suspended in solvent in such way that spots are above solvent surface.
5. After certain time the organic solvent will start to travel up the paper by capillary action and will drag the spots up at different rate.
6. When the solvent reached to the top of paper then paper is removed and left to dry.
7. The colour spots are identified easily while visualisation techniques are used to see the colourless spots. The distance travelled by substance from where they were spotted will depend upon their polarity
8. Filter paper will act stationary phase while solvent will act mobile phase



9. The Rf value is the retention factor for the compound and gives indication that how will be stationary phase retained the compound. The R_f (retention factor) is calculated by formula given below

$$R_f = \frac{\text{distance travelled by the spot}}{\text{distance travelled by the solvent front}}$$

Applications of paper chromatography:

- It is used to separate biological active system of acids, carbohydrates, amino acid etc
- It is used in the separation and analysis of various organic compounds
- It is used for the separation of some inorganic cations and radioisotopes.

Fill in the blanks

1. The most common types of porous medium used in the laboratory for filtration are filter paper and crucibles.
2. Filter paper is a suitable medium for separation of gelatinous precipitate.
3. In qualitative filter paper the ash should not exceed 0.0001g
4. the rubber tipped rod which is used to remove precipitate from the side of beaker in filtration is called police man