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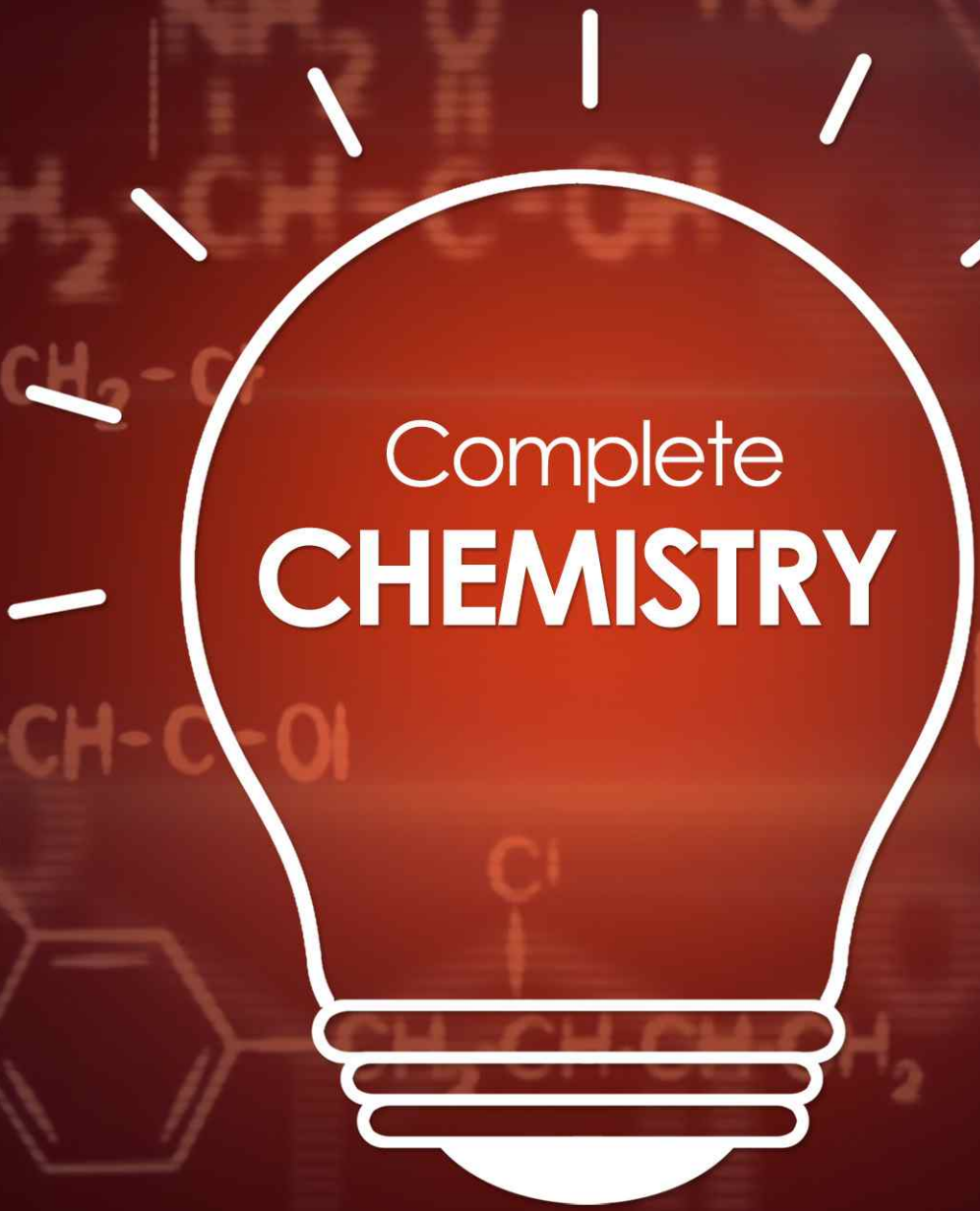


Complete CHEMISTRY

IIT-JEE · NEET · CBSE eBOOKS

CLASS 11&12th

CLASS 12th

Purification & Characterisation of Organic Compounds

01. Purification of Organic Compound

The methods to be employed depend on the physical state of the compound.

Crystallisation : This method is based on the difference in the solubility of the organic compound and its impurities in a solvent.

Preparation of the solution: Organic substance is powdered and is dissolved in a suitable solvent by heating. The amount of solvent should by just sufficient to dissolve the whole of the solid on heating.

Choice of Solvent : The choice of solvent is very important in the crystallisation process. The main conditions of the solvent are:

(i) The organic substance should dissolve in the solvent upon heating and it should get separated on cooling.

(ii) The solvent should not dissolve the impurities.

(iii) The solvent should not react chemically with the substance.

Sublimation : Certain organic solids directly change from solid to vapour state on heating. This process is called sublimation. The vapours on cooling change back to the solid form.

$$\text{Solid} \underset{\text{Cool}}{\overset{\text{Heat}}{\rightleftharpoons}} \text{Vapour}$$

The sublimation process is used for the separation of those solids which sublime on heating from non-volatile solids. The process is generally used for the purification of camphor, naphthalene, anthracene, benzoic acid, etc. containing non-volatile impurities.

Distillation : This method is used for the purification of liquids which boil without decomposition and contain non-volatile impurities.

Simple distillation involves its boiling point so that it is converted into vapours. On cooling the vapours, pure liquid is obtained. The distillate contains pure liquid while the impurities are left behind in the distillation flask e.g. Ether form ethyl alcohol.

Fractional Distillation : This process is used to separate a mixture of two or more miscible liquids which have boiling points close to each others. The fractionating column is a long tube provided with obstructions to the passage of the vapours moving upwards and liquid moving downwards. This method may be used to separate a mixture of acetone (b.p. 330K) and methyl alcohol (b.p. 338K).

Distillation under Reduced pressure (Vacuum Distillation) : Certain liquids have a tendency to decompose at a temperature below their normal boiling points.

Ex. glycerol boils with decomposition at 563K

Steam distillation : The process of steam distillation is used for the separation and purification of liquid which is appreciably volatile in steam, from non-volatile components of a mixture. Thus, the process of steam distillation is used to purify the substances which

(i) are volatile in steam but are not miscible with water

(ii) Possess sufficiently high vapour pressure at the boiling point temperature of water (100^0C)

(iii) Contain non-volatile impurities. The process of steam distillation can be applied for the separation of a mixture of m-nitrophenol and p-nitrophenol. In this process, water vapours carry along with them vapours of o-nitrophenol which is more volatile and they get condensed in the receiver; p-nitrophenol with higher b.p remains in the distillation flask. The method can also be used for the purification of impure sample of aniline.

Chromatography : This method is based on the differences in the rates at which the components of a mixture are adsorbed on a suitable adsorbent. There are many forms of chromatography such as column chromatography, paper chromatography, thin layer chromatography (TLC), gas chromatography, etc. The simplest method is column chromatography.

Applications of chromatographic method : This method has been used

(i) To separate ortho and para nitro-anilines.

(ii) To separate blue and red dyes.

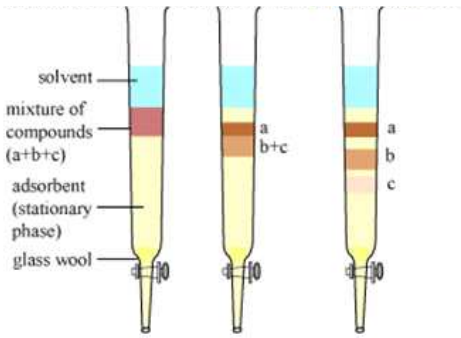
(iii) To separate and purify plant pigments and other natural products.

Types of chromatography :

Based on the principle involved chromatography is classified into different categories. Two of these are.

(i) **Adsorption chromatography :** Adsorption chromatography is based on the fact that different compounds are adsorbed on an adsorbent to different degrees. Commonly used adsorbents are silica gel and alumina, When a mobile phase is allowed to move over a stationary phase (adsorbent), the components of the mixture move by varying distances over the stationary phase. Following are two main types of chromatographic techniques based on the principle of differential adsorption.

- **Column chromatography :** Column chromatography involves separation of a mixture over a column of adsorbent (stationary phase) packed in a glass tube. The column is fitted with a stopcock at its lower end. The mixture adsorbed on adsorbent is placed on the top of the adsorbent column packed in a glass tube. An appropriate eluant which is a liquid or a mixture of liquids is allowed to flow down the column slowly. Depending upon the degree to which the compounds are adsorbed, complete separation takes place. The most readily adsorbed substances are retained near the top and others come down to various distances in the column.

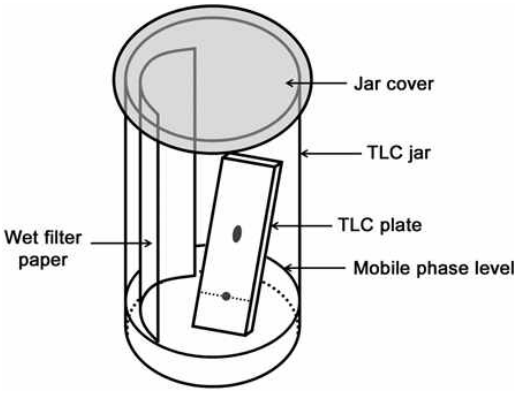


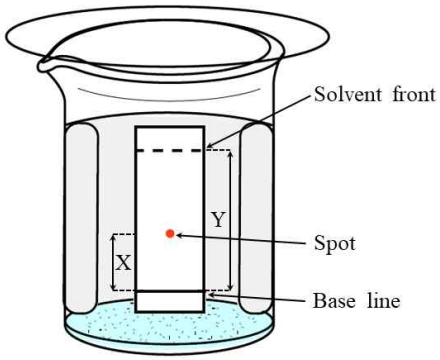
- **Thin layer chromatography :** Thin layer chromatography (TLC) is another type of adsorption chromatography, which involves separation of substances of a mixture over a thin layer of an adsorbent coated on glass plate. A thin layer (about 0.2 mm thick) of an adsorbent (silica gel or alumina) is spread over a glass plate of suitable size. The plate is known as thin layer chromatography plate or chromaplate. The solution of the mixture to be separated is applied as a small spot about 2 cm above one end of TLC plate. The glass plate is then placed in a closed jar containing the eluant. As the eluant rises up the plate, the components of the mixture move up along with the eluant to different distances depending on their degree of adsorption and separation takes place.

The relative adsorption of each component of the mixture is expressed in terms of its retardation factor.

i.e $R_f = \frac{\text{Distance moved by the substance from base line (x)}}{\text{Distance moved by the solvent from base line (y)}}$

The spots of coloured compounds are visible on TLC plate due to their original colour. The sport of colourless compounds which are invisible to the eye but fluoresce, can be detected by putting the plate under ultraviolet light.





(ii) **Partition Chromatography:**

Partition chromatography is based on continuous differential partitioning of components of mixture between stationary and mobile phase. Paper chromatography is a type of partition chromatography. In paper chromatography, a special quality paper known as chromatography paper is used. Chromatography paper contains water trapped in it, which acts as the stationary phase. A strip of chromatography paper spotted at the base with the solution of the mixture is suspended in a suitable solvent or a mixture of solvent. This 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 known as a chromatogram. The spots of the separated coloured compounds are visible at different heights from the position of intial spot on the chromatogram. The spots of the separated colourless compounds may be observed either under ultraviolet light or by the use of an appropriate spray regent as discussed under thin layer chromatography.