New NCERT Topic - Purification and Analysis of Organic Compound

METHODS OF PURIFICATION OF ORGANIC COMPOUNDS

Organic compounds obtained either from natural sources or synthesized in lab are contaminated with impurities.

Various methods are used for removal of impurities from an organic compound, depends on the nature of compound and type of impurities present in it. The following methods are commonly used for purification.

1. Sublimation
2. Crystallization
3. Distillation
4. Solvent extraction (differential extraction)
5. Chromatography

SUBLIMATION

- In sublimation, a solid is converted directly into gaseous state on heating and vice versa in case of cooling.

$$\text{Solid} \underset{\text{Cold}}{\overset{\Delta}{\rightleftharpoons}} \text{gaseous}$$

  For example, camphor, naphthalene, benzoic acid, iodine, ammonium chloride, anthracene, anthroquinone and phthallic anhydride are purified by sublimation.
- It is suitable for the solids which have non-volatile impurities and undergo sublimation on heating.

CRYSTALLIZATION

- Crystallization is based on the fact that during the crystal formation, impurities are left out as they are insoluble or feebly soluble in the used solvent. For crystallization, a suitable solvent like H_2O, R—OH, R—O—R, CCl_4, C_6H_6, acetone or petroleum are used. For example,
  (*a*) A mixture of benzoic acid and naphthalene is separated by hot water in which only benzoic acid dissolves.
  (*b*) The impurities of common salt in sugar can be removed by hot ethanol as only sugar dissolves in it.

Fractional Crystallization

- Fractional crystallization involves repeated crystalli zation.
- It is used when the substances differ slightly in the solubilities. For example, in a mixture of $KClO_3$ and KCl, KCl is more soluble than $KClO_3$ in cold water.

DISTILLATION

The vapourisation of a liquid by heating and subsequent condensation of vapours by cooling is known as distillation.

Distillation is an important method used to separate and its principle work on:

(*a*) Volatile liquids from non-volatile impurities.
(*b*) Liquids having sufficient difference in boiling points.

Distilaltion can be further divided into

Simple Distillation

The substances which do not decompose on heating at their boiling point and have non-volatile impurities are purified by this method. It is used when liquids differ in their boiling point by 30 – 50 K. For example

(*i*) Chloroform (B.P. 334 K) & Aniline (B.P. 457 K)
(*ii*) Ether (B.P. 308 K) & Toluene (B.P. 384 K)
(*iii*) Benzene (B.P. 353 K) & Aniline (B.P. 475 K)

Fractional Distillation

- Fractional distillation is used when the substances have nearly same boiling points (5°C to 10°C difference only). It involves repeated distillations and condensations.
  This process is carried out by using fractionating columns. Fractionating column is a special type of long glass tube provided with obstructions to the passage of the vapour upwards and that of liquid downwards.
- The more volatile liquid (with a lower boiling point) distils off before the less volatile liquid (with higher boiling point).

This method may be used to separate a mixture of acetone (b. p. 330 K) and methyl alcohol (b. p. 338 K) or a mixture of benzene and toluene. One of the technological applications of fractional distillation is to separate different fractions of crude oil in petroleum industry into various useful fractions such as gasoline, kerosene oil, diesel oil and lubricating oil etc.

Distillation Under Reduced Pressure (Vacuum Distillation)

- This method is used to purify liquids having very high boiling points, which decompose at or below their boiling points.

  It is based on the assumption that a liquid boils at a temperature when its V.P. becomes equal to that of the atmospheric pressure. It means if pressure is reduced distillation can occur below the Boiling Point of the substance.
- Here the pressure is reduced during distillation to avoid decomposition, so it is also called distillation at reduced pressure.
- Glycerine is purified by vacuum distillation.
- H_2O_2, formaldehyde are purified by vacuum distillation.
- Glycerol can be separated from spent-lye in soap industry by using vacuum distillation.
- Sugarcane juice is concentrated in sugar industry by evaporation under reduced pressure which saves lot of fuel.

Steam Distillation

- This method is used for separation and purification of organic compounds (solids or liquids) which
  (*a*) are steam volatile
  (*b*) are insoluble in water.
  (*c*) Possess high vapour pressure (10 – 15 mm of Hg at 373 K)
  (*d*) Contains non volatile impurities.
- Compounds which can be purified by steam distillation are aniline, nitrobenzene, bromobenzene, o-nitrophenol, o-hydroxy benzaldehyde (salicylaldehyde), o-hydroxy acetophenone, turpentine oil and essential oils.

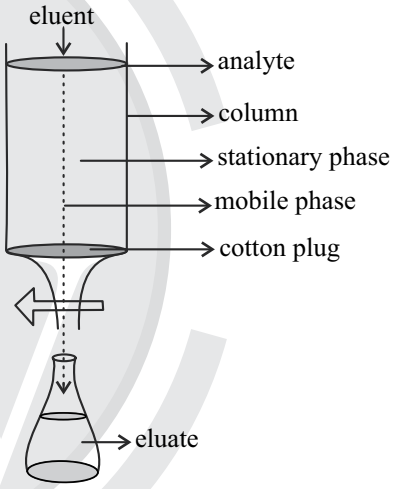
DIFFERENTIAL EXTRACTION

- The process of isolating an organic compound from its aqueous solution by shaking with a suitable solvent is called differential extraction. It is also called solvent extraction.
- When an organic compound is present in an aqueous medium, then it is separated by shaking it with an organic solvent in which it is more soluble than in water.
- Solvent should be immiscible with water and organic compound to be separated should be highly soluble in it.
- Organic solvent and aqueous solution are immiscible with each other, so they can form two distinct layers which can be separated by separatory funnel.
- Organic solvent is distilled or evaporated to get organic compound.
- Organic compound is less soluble in organic solvent so large quantity of solvent is required to extract small quantity of compound, which is said to be continuous extraction.
- Benzoic acid can be extracted from its aqueous solution using benzene as solvent.
- Ether is a better solvent in differential extraction due to:
  (*a*) Its less polarity
  (*b*) Least reactivity
  (*c*) Higher solubility of organic compounds

CHROMATOGRAPHY

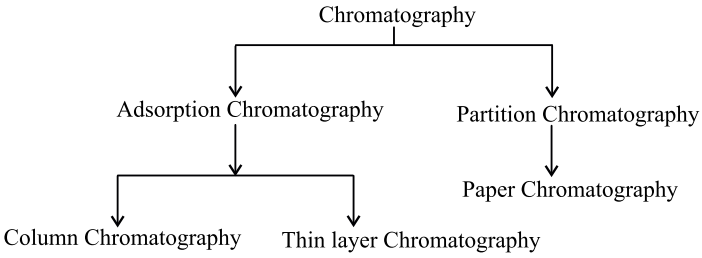
- Chromatography was introduced by Tswett in 1906.
- Here the separation and purification is carried out based on the difference in movement of components of a mixture through a stationary phase under the influence of a mobile phase.
- It is used for the separation of any substance which is present in a very small amount or which cannot be purified by other methods like distillation or crystallization.

some commonly used terms



Term	Definition
Mobile phase or carrier	solvent moving through the column
Stationary phase or adsorbent	substance that stays fixed inside the column
Eluant	fluid entering the column
Eluate	fluid exiting the column (that is collected in flask)
Elution	the process of washing out a compound through a column using a suitable solvent
Analyte	mixture whose individual components have to be separated and analyzed

Classification of Chromatography



Adsorption Chromatography

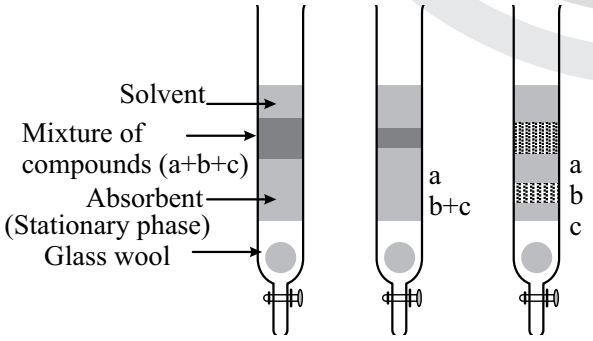
Adsorption chromatography is based on the fact that different compounds are adsorbed on an adsorbent to different degrees. Commonly used adsorbents are silica gel and alumina. When a mobile phase is allowed to move over a stationary phase (adsorbent), the components of the mixture move by varying distances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

(*a*) Column chromatography

(*b*) Thin layer chromatography.

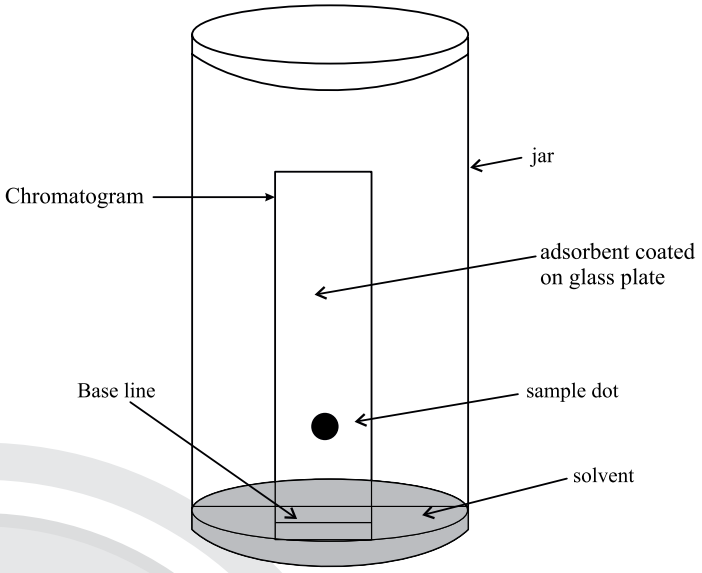
Column Chromatography

- 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.



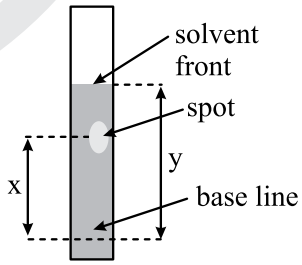
Thin Layer Chromatography

- 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 0.2mm thick) of an adsorbent (silica gel or alumina) is spread over a glass plate of suitable size.



Thin layer chromatography

- 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 the 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 value



Developed chromatogram

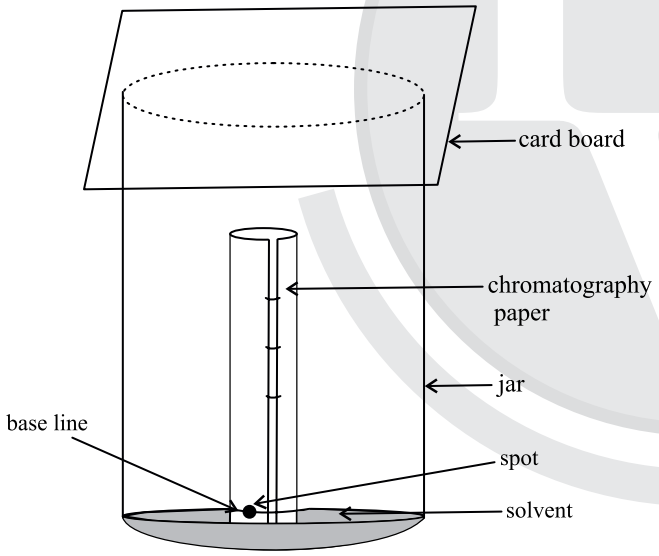
$$R_f = \frac{\text{Distance moved by the substance from base line } (x)}{\text{Distance moved by the solvent from base line } (y)}$$

- The spots of coloured compounds are visible on TLC plate due to their original colour.
- The spots of colourless compounds, can be detected by putting the plate under ultraviolet light.

- Another detection technique is to place the plate in a covered jar containing a few crystals of iodine. Spots of compounds, which adsorb iodine, will show up as brown spots.
- Sometimes an appropriate reagent may also be sprayed on the plate for example: Amino acids may be detected by spraying the plate with ninhydrin solution.

Partition Chromatography

- Partition chromatography is based on continuous differential partitioning of components of a mixture between stationary and mobile phases.
- Paper chromatography is a type of partition chromatography.
- In paper chromatography, a special quality paper known as chromatography paper (made of cellulose and water is absorbed in it) 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 solvents.
- This solvent acts as the mobile phase.
- The solvent rises up the paper by capillary action and flows over the spot.



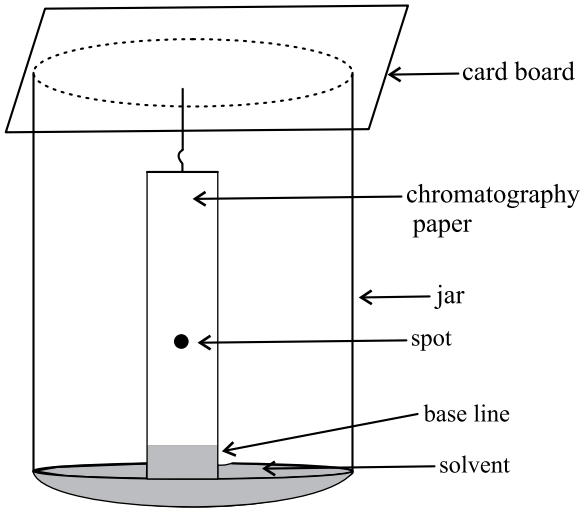


Fig : Paper chromatography (chromatography paper in two different shapes)

- 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 initial 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 reagent as discussed under thin layer chromatography.

Note:

- Main difference between TLC and Paper Chromatography are-

Thin Layer Chromatography	Paper Chromatography
It is based on Adsorption.	It is based on Partition
Stationary Phase → Glass Plate Coated with Silica gel	Stationary Phase → Water Trapped in the cellulose filter paper.
Corrosive reagent can be used.	Corrosive reagent can destroy the paper.

Train Your Brain

Example 1. Liquid which decompose below their normal boiling points can be distilled at lower temperature by-

(1) Increasing the pressure

(2) Decreasing the pressure

(3) Heating in water bath

(4) Heating in sand bath

Sol. (2) B.pt. gets lowered at low pressure.

Example 2. A bottle containing two immiscible liquids is given to you. They may be separated by using

(1) Fractionating coloumn

(2) Separating funnel

(3) Vacuum distillation

(4) Steam distillation

Sol. (2) Being immiscible liquids, they form different layers.

Concept Application

1. A mixture contains four solid organic compounds, *A*, *B*, *C* and *D*. On heating only C changes from solid to vapour state. *C* can be separated from the rest in the mixture by-

(1) Distillation (2) Sublimation

(3) Fractional distillation (4) Crystallisation

2. The best method to separate the mixture of ortho and para nitrophenol (1:1) is-

(1) Steam distillation (2) Crystallization

(3) Vapourisation (4) Colour spectrum

3. The separation of mixture of two compounds by chromatographic technique is based upon-

(1) Differential solubilities

(2) Different densities

(3) Different absorption

(4) Differential adsorption

QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS (DETECTION OF ELEMENTS)

Qualitative analysis is the analysis of the species present in a given compound. For example, if a compound is taken, the qualitative analysis would be more focused on finding the elements and the ions present in the compound rather than study as to how much they are present.

The qualitative analysis of an organic compound involves detection of all elements present in it.

DETECTION OF CARBON AND HYDROGEN

Carbon and hydrogen are detected by heating the compound with copper(II) oxide. Carbon present in the compound is oxidised to carbon dioxide (tested with lime-water, which develops turbidity) and hydrogen to water (tested with anhydrous copper sulphate, which turns blue).

$$C + 2CuO \xrightarrow{\Delta} 2Cu + CO_2$$

$$2H + CuO \xrightarrow{\Delta} Cu + H_2O$$

$$CO_2 + Ca(OH)_2 \longrightarrow CaCO_3 \downarrow + H_2O$$

$$\underset{\text{White}}{5H_2O + CuSO_4} \longrightarrow \underset{\text{Blue}}{CuSO_4 \cdot 5H_2O}$$

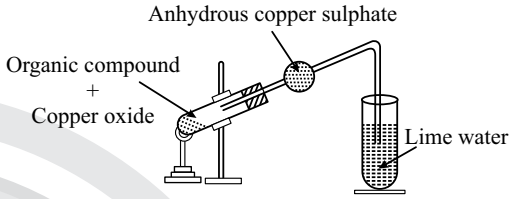


Fig. Detection of carbon and hydrogen in an organic compound

DETECTION OF OTHER ELEMENTS

Lassaigne's test

Nitrogen, sulphur, halogens and phosphorus present in an organic compound are detected by **"Lassaigne's test"**. The elements present in the compound are converted from covalent form into the ionic form by fusing the compound with sodium metal. Following reactions take place:

$$Na + C + N \xrightarrow{\Delta} NaCN$$

$$2Na + S \xrightarrow{\Delta} Na_2S$$

$$Na + X \xrightarrow{\Delta} Nax$$

C, N, S and X come from organic compound.

Cyanide, sulphide and halide of sodium so formed on sodium fusion are extracted from the fused mass by boiling it with distilled water. This extract is known as **sodium fusion extract**.

Test for Nitrogen

The sodium fusion extract is boiled with iron (II) sulphate and then acidified with concentrated sulphuric acid. The formation of Prussian blue colour confirms the presence of nitrogen. Sodium cyanide first reacts with iron (II) sulphate and forms sodium hexacyanoferrate(II). On heating with concentrated sulphuric acid some iron (II) ions are oxidised to iron (III) ions which react with sodium hexacyanoferrate(II) to produce iron(III) hexacyanoferrate(II) (ferriferrocyanide) which is Prussian blue in colour.

$$6CN^- + Fe^{2+} \longrightarrow [Fe(CN)_6]^{4-}$$

$$3[Fe(CN)_6]^{4-} + 4Fe^{3+} \xrightarrow{xH_2O} \underset{\text{Prussian blue}}{Fe_4[Fe(CN)_6]_3.xH_2O}$$

Test for Sulphur

(*i*) The sodium fusion extract is acidified with acetic acid and lead acetate is added to it. A black precipitate of lead sulphide indicates the presence of sulphur.

$S^{2-} + Pb^{2+} \longrightarrow \underset{\text{Black}}{PbS}$

(*ii*) On treating sodium fusion extract with sodium nitroprusside, appearance of a violet colour further indicates the presence of sulphur.

$S^{2-} + [Fe(CN)_5 NO]^{2-} \longrightarrow \underset{\text{Violet}}{[Fe(CN)_5NOS]^{4-}}$

In case, nitrogen and sulphur both are present in an organic compound, sodium thiocyanate is formed. It gives blood red colour and no Prussian blue since there are no free cyanide ions.

$Na + C + N + S \longrightarrow NaSCN$

$Fe^{3+} + SCN^- \longrightarrow \underset{\text{Blood red}}{[Fe(SCN)]^{2+} \text{ or } Fe(SCN)_3}$

If sodium fusion is carried out with excess of sodium, then thiocyanate decomposes to yield cyanide and sulphide. These ions give their usual tests.

$NaSCN + 2Na \longrightarrow NaCN + Na_2S$

Test for Halogens

(*i*) The sodium fusion extract is acidified with nitric acid and then treated with silver nitrate. A white precipitate, soluble in ammonium hydroxide shows the presence of chlorine, a yellowish precipitate; sparingly soluble in ammonium hydroxide shows the presence of bromine and a yellow precipitate, insoluble in ammonium hydroxide shows the presence of iodine.

$X^- + Ag^+ \longrightarrow AgX$

X represents a halogen **:**Cl, Br or I.

If nitrogen or sulphur is also present in the compound, the sodium fusion extract is first boiled with concentrated nitric acid to decompose cyanide or sulphide of sodium formed during Lassaigne's test. Otherwise these ions would interfere with silver nitrate test for halogens.

(*ii*) Beilstein's test

Organic compound $\xrightarrow[\text{Copper wire}]{\text{Heated over}}$ Green flame confirms the presence of halogn's in the organic compound

Test for Phosphorus

The compound is heated with an oxidising agent (sodium peroxide). The phosphorus present in the compound is oxidised to phosphate. The solution is boiled with nitric acid and then treated with ammonium molybdate. A yellow colouration or precipitate indicates the presence of phosphorus.

$Na_3PO_4 + 3HNO_3 \longrightarrow H_3PO_4 + 3NaNO_3$

$H_3PO_4 + 12(NH_4)_2 MoO_4 + 21HNO_3 \longrightarrow \underset{\text{Ammonium molybdate}}{}$

$(NH_4)_3 PO_4.\ 12MoO_3 + 21NH_4NO_3 + 12H_2O$
Ammonium phosphomolybdate

Test for Oxygen

There is no direct test for oxygen. If organic compound is heated in a dry test tube in nitrogen atmosphere, if water droplets are formed on the walls of the test tube indicates presence of oxygen. Presence of oxygen can be known by testing functional groups containing oxygen.

eg: —OH, —COOH, —CHO, —NO_2 etc.

Train Your Brain

Example 4. In Lassaigne's test for nitrogen, the blue colour is due to the formation of-

(1) Ferric ferrocyanide (2) Potassium ferrocyanide

(3) Sodium ferrocyanide (4) Sodium cyanide

Sol. (*a*) In Lassaigne's test substance is heated strongly with sodium metal then water extract is boiled with alkaline $FeSO_4$ solution and after cooling $FeCl_3$ solution and excess of HCl is added in it. If prussian blue or green ppt. is obtained then Nitrogen is confirmed-

$Na + C + N \longrightarrow NaCN$

$FeSO_4 + 2NaOH \longrightarrow Fe(OH)_2 + Na_2SO_4$

$6NaCN + Fe(OH)_2 \longrightarrow Na_4[Fe(CN)_6] + 2NaOH$

$\underset{\text{Sodium ferrocyanide}}{3Na_4[Fe(CN)_6]} + 4FeCl_3 \longrightarrow \underset{\text{prussian blue or green}}{Fe_4[Fe(CN)_6]_3} + 12NaCl$

Example 5. In sodium fusion test of organic compounds, the nitrogen of an organic compound is converted to-

(*a*) Sodamide (*b*) Sodium cyanide

(*c*) Sodium nitrite (*d*) Sodium nitrate

Sol. (*b*) $Na + C + N \xrightarrow{\text{Fusion}} NaCN$

Example 6. The Lassaigne's extract is boiled with conc. HNO_3 while testing for halogens. By doing so it:

(*a*) Decomposes Na_2S and NaCN, if formed.

(*b*) Helps in the precipitation of AgCl.

(*c*) Increases the solubility product of AgCl.

(*d*) Increases the concentration of NO_3 – ions.

Sol. (*a*) Sodium extract (Lassaigne's extract) is boiled with dilute HNO_3 before testing for halogens to decompose NaCN or Na_2S present in the Lassaigne's extract otherwise these will produce ppt. with $AgNO_3$ and would thus interfere with the test of halogens.

Concept Application

4. A compound which does not give a positive test in the Lassaigne's test for 'N' is-

(1) Glycine (2) Phenyl hydrazine

(3) Urea (4) Azobenzene

5. In Lassaigne's test, the organic compound is fused with sodium metal so as to-

(1) Burn the compound

(2) Form a sodium derivative

(3) Convert N, S, or halogen into soluble ionic compound

(4) None of these

6. Which of the following compound will give blood red colour while doing the Lassaigne's test for N.

(1) $(NH_2)_2C=O$ (2) $H_2N(C_6H_4)SO_3H$

(3) $C_6H_5SO_3H$ (4) $CHCl_3$

7. In a Lassaignes's test for sulphur in the organic compound with sodium nitroprusside solution the purple colour formed is due to-

(1) $Na_4[Fe(CN)_5NOS]$

(2) $Na_3[Fe(CN)_5S]$

(3) $Na_2[Fe(CN)_5NOS]$

(4) $Na_3[Fe(CN)_6]$

QUANTITATIVE ANALYSIS

Quantitative chemical analysis, branch of chemistry that deals with the determination of the amount or percentage of one or more constituents of a sample. A variety of methods is employed for quantitative analyses, which for convenience may be broadly classified as chemical or physical, depending upon which properties are utilized.

It involves the estimation of percentage composition of various elements by suitable methods.

ESTIMATION OF CARBON AND HYDROGEN

- Carbon and hydrogen are estimated by **Liebig's** combustion method.
- A known mass of an organic compound is burnt in the presence of excess of oxygen (free from CO_2) and Cupric oxide (CuO).
- Carbon and hydrogen present in the compound are oxidised to CO_2 and H_2O respectively.

$$C_xH_y+\left(x+\frac{y}{4}\right)O_2\xrightarrow{\Delta}xCO_2+\frac{y}{2}H_2O$$

- CO_2 and H_2O produced are weighed by absorbing in concentrated solution of potassium hydroxide and anhydrous calcium chloride (or) magnesium perchlorate respectively.

$$\%C=\frac{12}{44}\times\frac{\text{wight of } CO_2 \text{ formed}}{\text{weight of organic Compound}}\times100$$

$$\%H=\frac{2}{18}\times\frac{\text{wight of } H_2O \text{ formed}}{\text{weight of organic Compound}}\times100$$

ESTIMATION OF NITROGEN

Nitrogen present in organic compound is estimated by

(*a*) Dumas method

(*b*) Kjeldahl's method

Dumas Method

- In this method nitrogen present in the organic compound is converted into N_2.
- A weighed amount of organic compound is heated with cupric oxide in an atmosphere of carbondioxide.
- Carbon and hydrogen present in the compound are oxidised to CO_2 and H_2O, while N_2 is set free.
- Some oxides of nitrogen formed are reduced to free nitrogen by passing over heated copper gauze.

$$C_xH_yN_z+\left(2x+\frac{y}{2}\right)CuO\rightarrow xCO_2+\frac{y}{2}H_2O+\frac{z}{2}N_2+\left(2x+\frac{y}{2}\right)Cu$$

- Oxides of nitrogen + Cu $\rightarrow$ N_2 + CuO
- The mixture of gases produced is collected over caustic potash solution (KOH solution) which absorbs CO_2.

$$\%N=\frac{28}{22400}\times\frac{\text{Volume of nitrogen in ml at STP}}{\text{Weight of organic compound}}\times100$$

Kjeldahl's Method

- In this method nitrogen present in the organic compound is converted into ammonia (NH_3).
- A known mass of organic compound containing nitrogen is heated with concentrated sulphuric acid in presence of K_2SO_4 and $CuSO_4$ then nitrogen present in the compound is converted into ammonium sulphate.
- Organic compound + H_2SO_4 $\rightarrow$ $(NH_4)_2SO_4$
- The resulting solution is distilled with excess of sodium hydroxide.

  $(NH_4)_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2NH_3 + 2H_2O$
- Ammonia evolved is absorbed in a known but excess volume of standard HCl (or) H_2SO_4 solution.
- The acid left unreacted is estimated by titration against standard solution of sodium hydroxide.

  Percentage of Nitrogen =

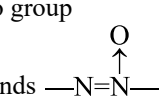
$$\frac{14}{1000}\times\frac{V\times N}{\text{wt. of organic compound}}\times100$$

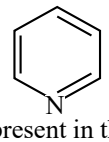
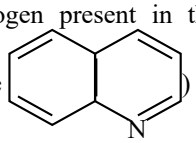
$$\%N=\frac{1.4\times V\times N}{\text{Weight of organic compound}}$$

Where V = Volume of acid in mL neutralised by ammonia

N = Normality of acid.

- This method is simpler and more convenient.
- It is mainly used to find percentage of nitrogen present in food stuffs, soils, fertilizers and various agricultural products.
- This method is not applicable to compounds containing nitro ($-NO_2$), Nitroso (NO), azo group

($-N = N-$), azoxy compounds 

and nitrogen present in the ring (pyridine  and quinoline ) because nitrogen present in these compounds is not quantitatively converted into ammonium sulphate.

- K_2SO_4 increases boiling point of H_2SO_4 and $CuSO_4$ acts as catalyst.

ESTIMATION OF HALOGENS (CARIUS METHOD)

- A weighed amount of an organic compound is heated with fuming nitric acid in the presence of silver nitrate contained in a hard glass tube known as carius tube.
- Carbon and hydrogen present in the compound is converted into CO_2 and H_2O.
- Halogen present in the organic compound is converted into silver halide.
- The precipitate is washed, dried and weighed.

Percentage of halogen =

$$\frac{\text{Atomic weight of halogen}}{\text{M.wt of silver halide}} \times \frac{\text{Weight of silver halide formed}}{\text{Weight of organic compound}} \times 100$$

$$\%Cl = \frac{35.5}{143.5} \times \frac{\text{w.t. of AgCl formed}}{\text{Wt. of organic compound}} \times 100$$

$$\%Br = \frac{80}{188} \times \frac{\text{wt. of AgBr formed}}{\text{Wt. of organic compound}} \times 100$$

$$\%I = \frac{127}{235} \times \frac{\text{wt. of AgI formed}}{\text{Wt. of organic compound}} \times 100$$

ESTIMATION OF SULPHUR (CARIUS METHOD)

- A weighed amount of organic compound is heated in a carius tube with sodium peroxide or fuming nitric acid.
- Sulphur present in the compound is oxidised into sulphuric acid, which is treated with $BaCl_2$ solution gives precipitate of $BaSO_4$.
- It is filtered, the precipitate is washed, dried and weighed.

$$\%S = \frac{\text{Atomic wt. of sulphur}}{\text{Mol. wt. of } BaSO_4} \times \frac{\text{wt. of } BaSO_4 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

$$\%S = \frac{32}{233} \times \frac{\text{wt. of } BaSO_4 \text{ formed}}{\text{wt. of organic compoud}} \times 100$$

ESTIMATION OF PHOSPHORUS (CARIUS METHOD)

A weighed amount of organic compound is heated with fuming nitric acid, then phosphorus present in the compound is oxidised to phosphoric acid.

- Phosphoric acid is precipitated as magnesium ammonium phosphate ($MgNH_4PO_4$), by addition of magnesia mixture ($MgCl_2 + NH_4OH + NH_4CI$)
- Magnesium ammonium phosphate is washed, dried and it is heated strongly to get magnesium pyrophosphate ($Mg_2P_2O_7$).

$$\underset{\text{(Phosphoric acid)}}{H_3PO_4} \xrightarrow{\text{Magnesia mixture}} \underset{\text{(Magnesium ammonium phosphate)}}{MgNH_4PO_4}$$

$$2MgNH_4PO_4 \xrightarrow{\Delta} \underset{\substack{\text{Magnesium}\\\text{Pyrophosphate}}}{Mg_2P_2O_7} + 2NH_3 + H_2O$$

$$\%P = \frac{62}{222} \times \frac{\text{wt. of } Mg_2P_2O_7 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

Using Ammonium Molybdate

- Phosphoric acid is precipitated as ammonium phospho molybdate $(NH_4)_3PO_4.12MoO_3$ by adding ammonia and ammonium molybdate.

(Molecular mass of $(NH_4)_3PO_4.12MoO_3$ = 1877)

$$\%P = \frac{31}{1877} \times \frac{\text{wt. of } (NH_4)_3PO_4.12MoO_3 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

ESTIMATION OF OXYGEN

- Usually percentage of oxygen in organic compound is determined by method of difference.

% of oxygen = 100 – (sum of the percentages of all other elements)

- Oxygen present in the organic compound is estimated by **Aluise's method**.
- A known amount of organic compound is subjected to pyrolysis in a stream of nitrogen.
- The mixture of gaseous products containg oxygen is passed over red-hot coke, then all the oxygen is converted into carbon monoxide.

compound $\xrightarrow{\Delta} O_2 +$ other gaseous products

$$2C + O_2 \xrightarrow{1373K} 2CO$$

- CO formed is quantitatively converted into CO_2 by passing over warm Iodine pentoxide (I_2O_5).

$5CO + I_2O_5 \rightarrow I_2 + 5CO_2$

The resulting gaseous mixture (CO_2 and I_2) is passed through potassium iodide solution, which absorbs iodine, and then passed over KOH to absorb CO_2.

$$\% \text{ of oxygen} = \frac{16}{44} \times \frac{\text{wt. of } CO_2 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

Train Your Brain

Example 7. Liebig method is used for the estimation of

(1) Nirogen
(2) Sulphur
(3) Carbon and Hydrogen
(4) Halogens

Sol. (3) Liebig method is used for the estimation of carbon and hydrogen.

Example 8. The quantitative determination of halogen in an organic compound is known as-

(1) Dumas method
(2) Carius method
(3) Kjeldahl method
(4) Leibig method

Sol. (2) The quantitative determination of halogen in an organic compound is called as carius method.

Example 9. 0.2475 g of an organic compound gave on combustion 0.4950 g of carbon dioxide and 0.2025 g of water. The percentage of carbon and hydrogen are-

(1) 54.54, 9.09
(2) 52.54, 8.09
(3) 120, 5.8
(4) None of these

Sol. (1) Wt. of organic compound = 0.2475 g

Wt. of CO_2 produced = 0.4950 g

Wt. of H_2O produced = 0.2025 g

Percentage of carbon

$$= \frac{12}{44} \times \frac{\text{Wt. of } CO_2}{\text{Wt. of compound}} \times 100$$

$$= \frac{12}{44} \times \frac{0.4950}{0.2475} \times 100 = 54.54$$

Percentage of hydrogen

$$= \frac{2}{18} \times \frac{\text{Wt. of } H_2O}{\text{Wt of compound}} \times 100$$

$$= \frac{2}{18} \times \frac{0.2025}{0.2475} \times 100 = 9.09$$

Concept Application

8. Duma's method involves the determination of content of nitrogen in the organic compound in the form of -
(1) Gaseous NH_3 (2) Gaseous N_2
(3) NaCN (4) $(NH4)_2\ SO_4$

9. 0.59 g of an organic substance when treated with caustic soda evolved ammonia, which required 20 c.c. of N/2 sulphuric acid for neutralization. The percentage of nitrogen is-
(1) 40% (2) 53.6%
(3) 63.6% (4) 23.73%

10. If 0.2 gram of an organic compound containing carbon, Hydrogen and oxygen on combustion, yielded 0.147 gram carbon dioxide and 0.12 gram water. What will be the content of oxygen in the substance ?
(1) 73.29 % (2) 78.45 %
(3) 83.23 % (4) 89.50 %

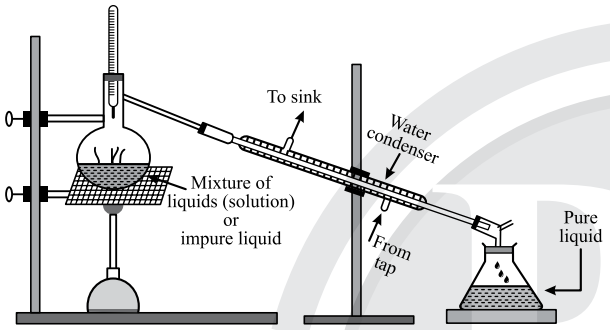
Crystallisation: It is based on the difference in the solubilities of the compound and the impurities in a suitable solvent.

Fractional Crystallization: Difference in solubilities of organic compounds and impurities are very less.

eg. urea and copper sulphate.

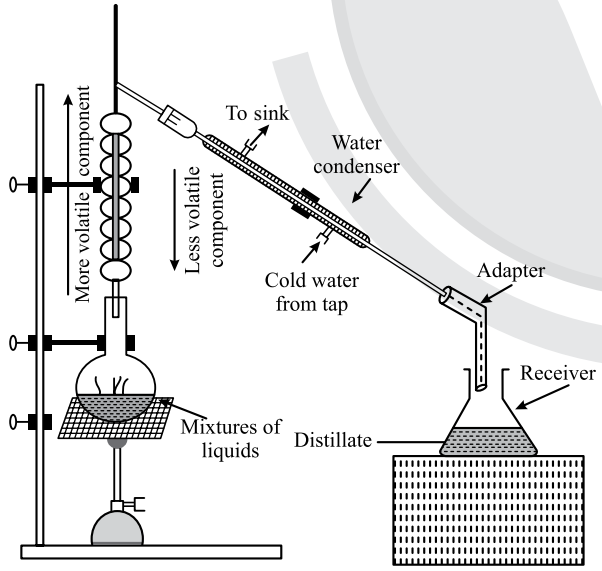
Distillation

(*i*) **Simple distillation:** Difference in boiling points of compounds is more than 40°C. e.g. chloroform (b. p. 334K) and aniline.



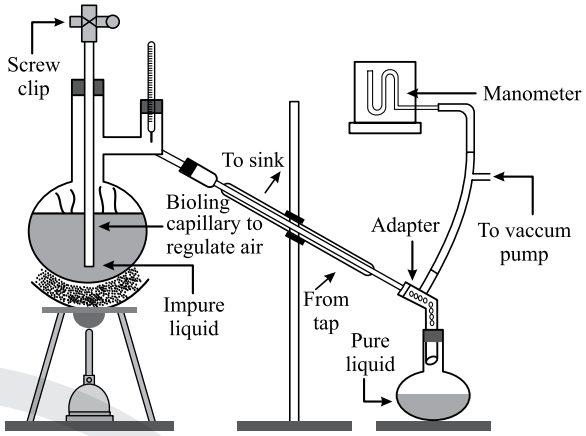
Simple distillation

(*ii*) **Fractional distillation:** Difference in boiling points of compounds is very less (5°C to 10°C). e.g. - acetone (b. p. 329K) and methyl alcohol (b. p. 338K).



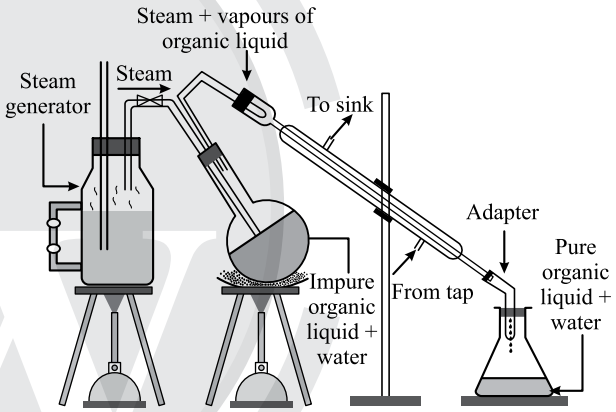
Fractional distillation

(*iii*) **Vacuum distillation:** Used for organic compounds which decompose at or below their boiling points. e.g. Glycerol.



Distillation under reduced pressure

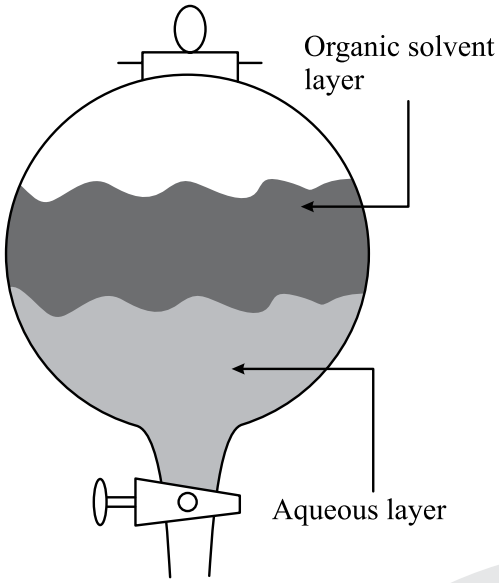
(*iv*) **Steam distillation**: Used for organic compounds which are immiscible with water and are steam volatile. e.g. Aniline.



Apparatus for steam distillation

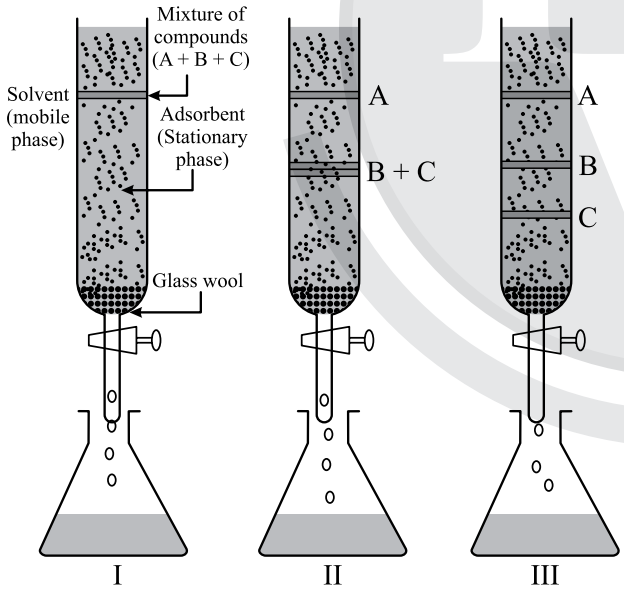
Differential extraction: Used to extract pure organic compounds from their aqueous solution by shaking with organic solvent in which they are highly soluble.

e.g. Benzoic acid from its aqueous solution using benzene.



Chromatography

- Used to purify small samples.
- Based on selective adsorption or partition between stationary and mobile phase.
- Column Chromatographybased on adsorption, used for bulk quantities.
- Thin layer Chromatographybased on adsorption, used for quantitative analysis.



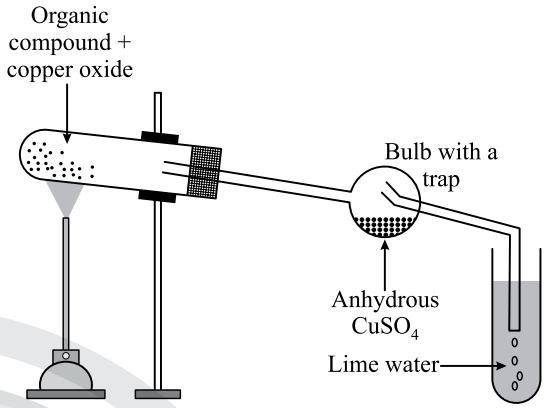
Column chromatography-stage I, II and III represent the progressive separation of the mixture into three bands

- Paper Chromatography- based on partition and used for quantitative and qualitative analysis.

QUALITATIVE ANALYSIS

Detection of C, H, N, halogens P, S, and oxygen.

Detection of Carbon and Hydrogen



Detection of carbon and hydrogen

$$\text{Organic Compound} + CuO \xrightarrow{\Delta} \underset{\text{Carbon dioxide}}{CO_2} + \underset{\text{Water}}{H_2O} + \underset{\text{Copper}}{Cu}$$

Passed into lime water $Ca(OH)_2$ → Lime water turns milky → Carbon (C) present In organic compound

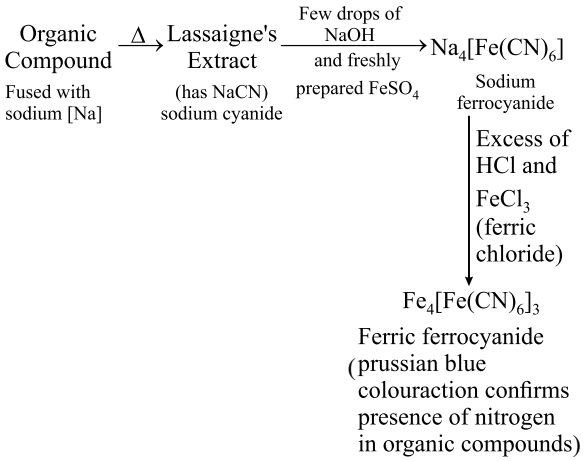
Passed into anhydrous $CuSO_4$ → Copper Sulphate turns blue → Hydrogen (H) present in Organic Compound

Detection of Nitrogen

$$\text{Organic Compound} + \underbrace{NaOH + CaO}_{\text{Sodalime}} \xrightarrow{\Delta} \text{Smell of } NH_3 \rightarrow \text{Confirms the presence of nitrogen in organic compound}$$

Lassaigne's Extract

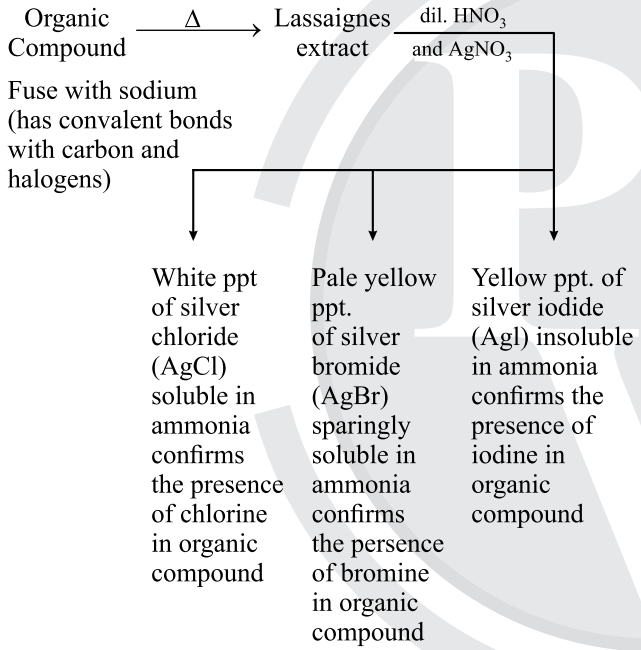
Lassaigne's extract is prepared to convert covalency of organic compound into electrovalency by fusing with Na.



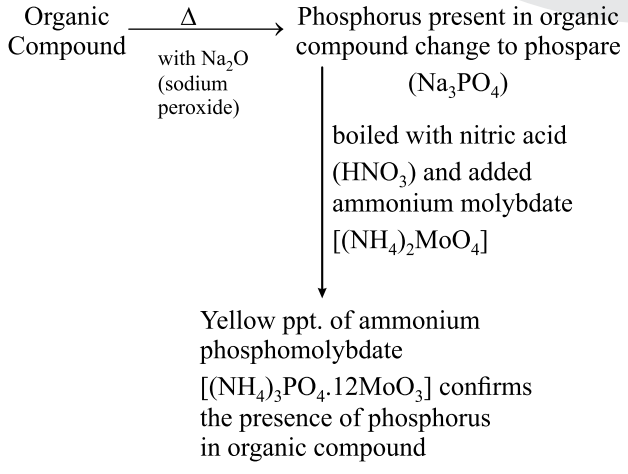
Detection of Halogen

Beilstein's test

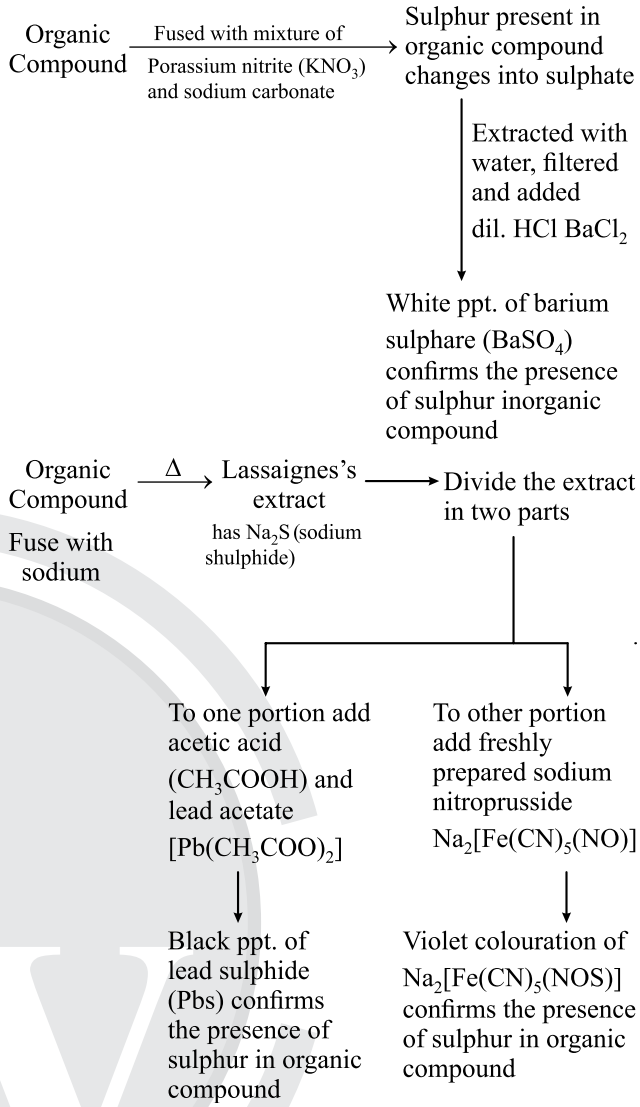
Organic compound $\xrightarrow[\text{Copper wire}]{\text{Heated over}}$ Green flame confirms the presence of halogn's in the organic compound



Detection of Phosphorus



Detection of Sulphur



Detection of Oxygen

Presence of oxygen in organic compound is detected by testing for functional group containing oxygen e.g. alcohol (–OH), aldehyde (–CHO), ketone (RCOR), carboxylic acid (–COOH), ester (–COOR) and nitro ($-NO_2$).

Quantitative Analysis

$$\%C = \frac{12}{44} \times \frac{\text{wt. of } CO_2}{\text{Wt. of org. compound}}$$

$$\%H = \frac{2}{18} \times \frac{\text{wt. of } H_2O}{\text{Wt. of org. compound}} \times 100$$

$$\%N = \frac{28}{22400} \times \frac{\text{Vol. of } N_2 \text{ at STP}}{\text{Wt. of org. comp.}} \times 100$$

or

$$\%N = \frac{\text{Volume of } N_2 \text{ at STP}}{8 \times \text{Wt. of org. compound}}$$ (Duma's method)

$$(\%N) = \frac{1.4 \times N \times V}{\text{wt. of org. compound}} \text{ (Kjeldhal's method)}$$

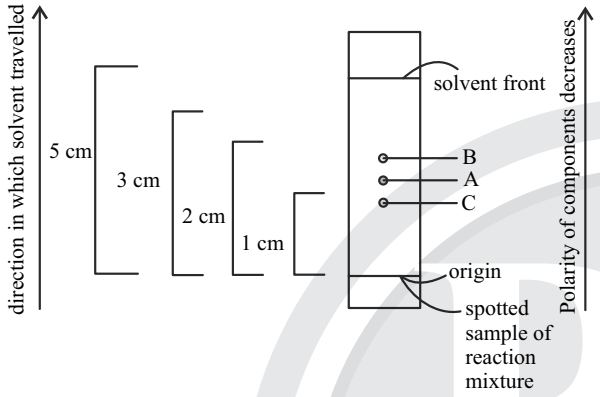
$$\%X = \frac{\text{At. wt. of X}}{\text{Mol. wt. AgX}} \times \frac{\text{Wt. of AgX}}{\text{wt. of org. compound}}$$

$$\%S = \frac{32}{233} \times \frac{\text{Wt. } BaSO_4}{\text{wt. of org. compound}} \times 100$$

$$\%P = \frac{62}{222} \times \frac{\text{Mass of } Mg_2P_2O_7}{\text{Wt. of org. compound}} \times 100$$

AARAMBH (SOLVED EXAMPLES)

1.



So just by looking at a TLC plate, can you tell which component is more polar and which component is less polar.

Sol. The relative adsorption of each component of the mixture is expressed in terms of its retardation factor i.e. R_f value.

$$R_f = \frac{\text{Distance moved by the substance from base line}}{\text{Distance moved by the solvent from base line}}$$

'Lower the R_f value, more polar the component.'

Component	Distance travelled by the component (cm)	Distance travelled by the solvent (cm)	retention factor (R_f)
C	1	5	$R_f = 1/5 = 0.2$
A	2	5	$R_f = 2/5 = 0.4$
B	3	5	$R_f = 3/5 = 0.6$

Based on the R_f, values (as calculated above), component C is the most polar and component B is the least polar.

2. 0.25g of an organic compound gave 30 cm^3 of moist dinitrogen at 288 K and 745 mm pressure. Calculate the percentage of nitrogen. (Aqueous tension at 288 K = 12.7 mm)

Sol. Mass of the substance = 0.25 g

Volume of moist dinitrogen = 30 cm^3

Temperature = 288K, Pressure 745 – 12.7 = 732.3 mm

Volume of dinitrogen at STP:

$$V_2 = \frac{P_1V_1}{T_1} \times \frac{T_2}{P_2} = \frac{732.3 \times 30 \times 273}{288 \times 760} = 27.4 \text{ cm}^3$$

Percentage of nitrogen in organic compound =

$$\frac{28}{22400} \times \frac{\text{vol. of } N_2 \text{ at STP}}{\text{wt. of organic compound}} \times 100$$

$$\frac{28}{22400} \times \frac{27.4}{0.25} \times 100 = 13.6$$

3. In Kjeldahl's estimation of nitrogen, the ammonia evolved from 0.5 g of an organic compound neutralised by 10 ml of 1M H_2SO_4. Calculate the percentage of nitrogen in the compound.

Sol. 10 ml of 1M H_2SO_4 = 20 ml of M NH_3

1000 ml of 1M ammonia contains 14 g nitrogen

20 ml of 1M ammonia contains $\frac{14 \times 20}{1000}$ g

$$\% \text{ of nitrogen} = \frac{14 \times 20 \times 100}{1000 \times 0.5} = 56.0$$

4. In Kjeldahl's estimation of nitrogen, the ammonia obtained from 0.5 g of an organic substance was passed into 100 cm^3 of $\frac{M}{10} H_2SO_4$. The excess of acid required 154 cm^3 of $\frac{M}{10}$ NaOH for neutralisation. Calculate the percentage of nitrogen in the compound.

Sol. m eq. of H_2SO_4 taken = molarity x basicity x volume (ml)

$$= \frac{1}{10} \times 2 \times 100 = 20$$

$$\text{m.eq of NaOH} = \frac{1}{10} \times 1 \times 154 = 15.4$$

m. eq of H_2SO_4 unused = 20 – 15.4 = 4.6

% of nitrogen =

$$\frac{1.4 \times \text{m.eq. of } H_2SO_4 \text{ unused}}{\text{wt. of organic compound}} = \frac{1.4 \times 4.6}{0.5} = 12.88$$

5. In Carius method, 0.1890 g of an organic compound gave 0.2870 g of silver chloride. Calculate the percentage of chlorine in the compound.

Sol. Weight of subtance = 0.1890 g

Weight of silver chloride = 0.2870 g

$$\% \text{ of chlorine} = \frac{\text{weight of AgCl} \times 35.5 \times 100}{\text{weight of subtance} \times 143.5}$$

$$= \frac{0.2870 \times 35.5 \times 100}{0.1890 \times 143.5} = 37.8$$

6. One gram of a bromoalkane on heating with excess silver nitrate in Carius tube method gave 0.94 g of yellow precipitate. What is the percent weight of halogen?

Sol. Weight of subtance = 1 g, Yellow precipitate is AgBr; Weight of AgBr = 0.94 g

$$\% \text{ of bromine} = \frac{0.94 \times 80 \times 100}{1 \times 188} = 40$$

7. In sulphur estimation, 0.157 g of an organic compound gave 0.4813 g of barium sulphate. What is the percentage of sulphur in the compound?

Sol. 233 g $BaSO_4$ contain 32 g sulphur

0.4813 g $BaSO_4$ contain $\frac{32 \times 0.4813}{233}$ g sulphur

$$\% \text{ of suphur} = \frac{32 \times 0.4813 \times 100}{233 \times 0.157} = 42.10$$

8. 0.2 g of an organic compound on analysis gave 0.147 g of carbondioxide, 0.12 g of water and 74.6 c.c of nitrogen at S.T.P. Calculate the weight percentages of constituents.

Sol. Weight of compound = W = 0.2 g

Weight of $CO_2 = W_1 = 0.147$ g

Weight of $H_2O = W_2 = 0.12$ g

Volume of N_2 at STP = 74.6 c.c.

$$\% \text{ of carbon} = \frac{W_1 \times 12 \times 100}{W \times 44} = \frac{0.147 \times 12 \times 100}{0.2 \times 44} = 20.04\%$$

$$\% \text{ of hydrogen} = \frac{W_2 \times 2 \times 100}{W \times 18} = \frac{0.12 \times 2 \times 100}{0.2 \times 18} = 6.66\%$$

$$\% \text{ of nitrogen} = \frac{V_2 \times 28 \times 100}{W \times 22400} = \frac{74.6}{8 \times 0.2} = 46.63\%$$

Remaining is oxygen.

% of oxygen = 100 – (%C + %H + %N)

= 100-73.33= 26.67%

9. 0.257 g of an organic substance was heated with conc H_2SO_4 and then distilled with excess of strong alkali. The ammonia gas evolved was absorbed in 50 ml of N/10 HCl which required 23.2 ml of N/10 NaOH for neutralisation at the end of the process. The percentage of nitrogen in the compound is-

(1) 14.6 (2) 18.0

(3) 17.0 (4) 15.5

Sol. (1) Volume of $\frac{N}{10}$ HCl taken = 50 ml

Volume of $\frac{N}{10}$ NaOH used for neutralisation of unused acid = 23.2 ml

Now N_1V_1 (NaOH) = N_2V_2(HCl)

23.2 ml of $\frac{N}{10}$ NaOH = 23.2 ml of $\frac{N}{10}$ HCl

$\therefore$ Volume of $\frac{N}{10}$ HCl unused = 23.2 ml

$\therefore$ Volume of $\frac{N}{10}$ HCl required for neutralization of NH_3

= 50 – 23.2 = 26.8 ml

26.8 ml of $\frac{N}{10}$ HCl = 26.8 ml of $\frac{N}{10}$ NH_3

1000 ml of 1 N NH_3 solution contains nitrogen = 14 g

26.8 ml of NH_3 solution contains nitrogen

$$= \frac{14 \times 26.8}{10 \times 1000}$$

Percentage of nitrogen

$$= \frac{14 \times 26.8 \times 100}{10 \times 1000 \times 0.257} = 14.6\%$$

10. Hydrazine and diazonium salts do not give Lassaigne's test. Why?

Sol. In the Lassaigne's test, nitrogen is converted to cyanide by combining with carbon of the compound. Hydrazine does not contain carbon and hence, cyanide cannot be formed.

Diazonium salts are unstable and lose nitrogen as N_2 gas on heating. Hence during fusion, no sodium cyanide is formed in Lassaigne's extract due to the loss of nitrogen.

11. A certain organic compound was found to contain 33.8% C, 5.6% H and 49.6% chlorine. Find the empirical formula of the organic compound.

Sol.

Element	%	Atomic ratio	Simplest ratio
C	33.8	33.8/12=2.8	2.8/0.7 . 4
H	5.6	5.6/1 = 5.6	5.6/0.7 = 8
Cl	49.6	49.6/35.5 =1.4	1.4/0.7=2
O	11.0	11/16 = 0.7	0.7/0.7 = 1

Thus, $C_4H_8Cl_2O$ is the empirical formula of the compound.

12. A well known antibiotic penicillin, contains, 57.49 % C, 5.39% H, 8.39% N, 9.58% S and the remainder being oxygen. Given that penicillin contains one atom of sulphur per molecule, calculate the molecular formula of the antibiotic.

Sol. % C = 57.49

% H = 5.39

% N = 8.39

% S = 9.58

% O = 100–(57.49 + 5.39 + 8.39 + 9.58)

= 19.15

Element	%	Atomic ratio	Simplest ratio
C	57.49	57.49/12=4.79	16
H	5.39	5.39/1 = 5.39	18
N	8.39	8.39/14=0.599	2
S	9.58	9.58/32 = 0.299	1
O	19.15	19.15/16=1.196	4

Empirical formula is $C_{16}H_{18}N_2SO_4$

As the given antibiotic contains only one atom of sulphur per molecule. The calculated empirical formula $C_{16}H_{18}N_2SO_4$, contains only one atom of sulphur Hence, the same is the molecular formula of the antibiotic.

13. Gas chromatography can be performed in X, whereas liquid chromatography can be performed in Y. Identify X and Y.

(1) X = only plane surfaces, Y = only columns

(2) X = only columns, Y = only plane surfaces

(3) X = only columns, Y = columns or plane surfaces

(4) X = columns or plane surfaces, Y = only plane surfaces

Sol. (3) Explanation: Gas chromatography can be done only in columns because in this method, the sample is vaporized and injected onto the top of the chromatographic column. It could be gas-liquid or gas-solid chromatography. Liquid chromatography has liquid as the mobile phase where sample ions or molecules are dissolved. So, it can be carried out either in a column or a plane.

14. Which is the most suitable carrier gas in gas chromatography?

(1) Helium (2) Nitrogen

(3) Oxygen (4) Carbon dioxide

Sol. (1) Explanation: In gas chromatography, the carrier gas should be an inert gas which does not react with the sample. Even though nitrogen and some other gases are also used, 90% of the instruments use helium as the carrier gas. Hydrogen is preferred for improved separations.

15. Which of the following is the apparatus for differential extraction?

(1) Separatory funnel (2) Porous sheet

(3) Packed column (4) Electric motor

Sol. (1) Explanation: A separatory funnel is used in differential extraction. Differential extraction involves separating the immiscible organic compound from the aqueous solvent. The separating funnel aids in this process of separating immiscible liquids. It also helps in layer formation, with the denser solvent at the bottom and the other on top. This layer formation is important for the process, and thus, separatory funnel is chosen as the apparatus for differential extraction.

SINGLE CHOICE TYPE QUESTIONS

1. How will you separate a solution (miscible) of benzene and $CHCl_3$?
(1) Sublimation (2) Filtration
(3) Distillation (4) Crystallisation

2. The best method for the separation of naphthalene and benzoic acid from their mixture is:
(1) Chromatography (2) Crystallisation
(3) Distillation (4) Sublimation

3. A mixture of camphor and KCl can be separated by:
(1) Evaporation (2) Sublimation
(3) Filtration (4) Decantation

4. Lassaigne's test in qualitative analysis is used to detect:
(1) Nitrogen (2) Sulphur
(3) Chlorine (4) All of these

5. Presence of halogen in organic compounds can be detected by using:
(1) Leibig's test (2) Duma's test
(3) Kjeldahl's test (4) Beilstein test

6. Medium of sodium extract is:
(1) Neutral
(2) Basic
(3) Acidic
(4) Depends on organic compound

7. A mixture of calcium sulphate and camphor can be separated by-
(1) Filtration (2) Evaporation
(3) Sublimation (4) Chromatography

8. In Carius method halogens are estimated as:
(1) X_2 (2) BaX_2
(3) PbX_2 (4) AgX

ASSERTION AND REASON TYPE QUESTIONS

Directions: These questions consist of two statements each, printed as Assertion and Reason. While answering these questions, you are required to choose any one of the following four responses.

(1) Both Assertion and Reason are True and the Reason is a correct explanation of the Assertion.
(2) Both Assertion and Reason are True but Reason is not a correct explanation of the Assertion.
(3) Assertion is True but the Reason is False.
(4) Assertion is False but Reason is True.

1. Assertion: Simple distillation can help in separating a mixture of propan-1-ol (boiling point 97°C) and propanone (boiling point 56°C).

Reason: Liquids with a difference of more than 20°C in their boiling points can be separated by simple distillation.

2. Assertion: Sulphur present in an organic compound can be estimated quantitatively by Carius method in which it can be oxidised to H_2SO_4.

Reason: Sulphur is separated easily from other atoms in the molecule and gets precipitated as light yellow solid.

MATCH THE COLUMN TYPE QUESTIONS

1. Match the type of mixture of compounds in Column-I with the technique of separation/purification given in Column-II.

	Column-I		Column-II
A.	Two solids which have different solubilities in a solvent and which do not undergo reaction when dissolved in it.	P.	Steam distillation
B.	Liquid that decomposes at its boiling point	Q.	Fractional distillation
C.	Steam volatile liquid	R.	Simple distillation
D.	Two liquids which have boiling points close to each other	S.	Distillation under reduced pressure
E.	Two liquids with large difference in boiling points.	T.	Crystallisation

(1) A-(T); B-(S); C-(P); D-(Q): E-(R)
(2) A-(Q); B-(R); C-(P); D-(S); E-(T)
(3) A-(T); B-(S); C-(P); D-(R); E-(Q)
(4) A-(R); B-(S); C-(T); D-(P); E-(Q)

2. Match the Column-I with Column-II.

	Column-I		Column-II
A.	Dumas Method	P.	Fuming nitric acid
B.	Kjeldahl's method	Q.	Excess CuO
C.	Estimation of Sulphur	R.	Fuming nitric acid + Silver nitrate
D.	Estimation of Halogens	S.	Conc. H_2SO_4

(1) A-(Q); B-(S); C-(P); D-(R)
(2) A-(P); B-(Q); C-(R); D-(S)
(3) A-(Q); B-(P); C-(S); D-(R)
(4) A-(S); B-(R); C-(Q); D-(P)

SHORT ANSWER TYPE QUESTIONS

1. Why is the fusion of organic compounds with sodium employed in qualitative tests for nitrogen, halogens, and sulfur? Explain how this process transforms these elements into more detectable forms and its significance in organic compound analysis.

2. (*a*) When dealing with a compound in a liquid state that decomposes at or below its boiling point, which purification method is better suited for the task?
   (2) Describe the procedure for separating a mixture containing ammonium chloride and common salt.
3. A 0.12 g sample of an organic compound containing phosphorus was subjected to standard analysis, yielding 0.22 g of $Mg_2P_2O_7$. Determine the compound's phosphorus content as a percentage?
4. Using the Carius method, 0.15 g of an organic compound produced 0.12 g of AgBr, calculate the percentage of bromine (Br) in the compound.
5. What are the key distinctions between the processes of filtration and evaporation when it comes to separating components from a liquid mixture?.
6. What methods or procedures can be employed to identify the existence of nitrogen and sulfur in Lassaigne's extract.

LONG ANSWER TYPE QUESTIONS

1. (1) What is the extract from Lassaigne? Will NaCN result in a nitrogen Lassaigne test being positive?
   (2) Which colour, in the case of a chemical containing both nitrogen and sulphur, will show up in the Lassaigne's test.
   (3) Why is distilled water used to create Lassaigne's extract? Can the Lassaigne test identify oxygen in a compound?
2. (1) Explain the fundamental principle underlying the determination of nitrogen content using the Dumas method.
   (2) A sample of an organic compound weighing 0.30 g produced 50 cm^3 of nitrogen gas under specific conditions of temperature and pressure (300 K and 715 mm Hg). Calculate the percentage of nitrogen in the compound, considering the vapor pressure of water at 300 K (15 mm Hg).
3. (1) What is the Carius method, and how is it used to determine the halogen content in an organic compound?
   (2) Determine the percentage of nitrogen (N) in a 0.25 g sample of an organic compound, given that it produced 38 cm^3 of nitrogen gas at 300 K and 96 kPa pressure.
4. Discuss the concept of fractional distillation and how it differs from simple distillation. Provide an example of a mixture that can be effectively separated using fractional distillation and explain the role of fractional distillation in achieving this separation.
5. (*a*) "During the estimation of nitrogen present in an organic compound by Kjeldahl's method, the ammonia evolved from 0.5 grams of the compound neutralized 10 mL of 1 M H_2SO_4. Calculate the percentage of nitrogen in the compound.
   (2) Explain the basic principle of chromatography and provide an example of its application in separating and analyzing different compounds.

CASE BASED STUDY QUESTIONS

1. **Read the following passage and answer the questions that follow:**

   In the realm of organic chemistry, the purification of organic compounds stands as a pivotal process, indispensable for obtaining substances of high purity. This methodical purification aids in removing impurities, unreacted reagents, and by-products, ensuring the reliability of subsequent experiments and the precision of analytical data. One commonly employed technique is recrystallization, where a solute is dissolved in a suitable solvent at an elevated temperature and then allowed to slowly crystallize as the solution cools, effectively separating the target compound from impurities. Fractional distillation is another invaluable method, primarily used for separating liquid mixtures with distinct boiling points. In chromatography, the separation relies on differential adsorption, making it versatile for complex mixtures. Lastly, sublimation, a unique purification process, involves the direct conversion of a solid into vapor without passing through the liquid phase, purifying compounds with high volatility.

   1. What is the primary purpose of the methods of purification of organic compounds?
      (1) To increase the quantity of the organic compound
      (2) To isolate impurities for further analysis
      (3) To obtain substances of high purity
      (4) To create new organic compounds
   2. Which method involves the dissolution of a solute in a solvent at an elevated temperature, followed by slow cooling to induce crystallization?
      (1) Sublimation (2) Fractional distillation
      (3) Chromatography (4) Recrystallization
   3. Fractional distillation is most suitable for separating:
      (1) Solid mixtures
      (2) Gaseous mixtures
      (3) Liquid mixtures with distinct boiling points
      (4) Solutions of organic compounds
   4. In chromatography, separation is primarily based on:
      (1) Boiling point differences
      (2) Solubility differences
      (3) Color differences
      (4) Differential adsorption

PURIFICATION AND SEPARATION OF ORGANIC COMPOUNDS

1. Sublimation is process where a solid-
(1) Melts
(2) Changes into liquid form.
(3) Boils
(4) Changes into vapour form directly.

2. A mixture of acetone and methanol can be separated by-
(1) Vaccum distillation (2) Steam distillation
(3) Fractional distillation (4) None of these

3. Turpentine oil can be purfied by-
(1) Vaccum distillation (2) Fractional distilliation
(3) Steam distillation (4) Simple distillation

4. Chromatography is used for the purification of-
(1) Solids (2) Gases
(3) Liquids (4) All of these

5. Steam distillation is applied to those organic compounds which are steam volatile and:
(1) Soluble in water.
(2) Insoluble is water.
(3) Sparingly soluble in water.
(4) Insoluble in all solvents.

6. Distillation involves all the following processes except:
(1) Change of state (2) Boiling
(3) Condensation (4) Evaporation

7. Turpentine oil and essential oil can be purified by:
(1) Fractional distillation (2) Steam distillation
(3) Vacuum distillation (4) Simple distillation

8. Chromatography technique is used for the separation of:
(1) Small samples of mixtures
(2) Plant pigments
(3) Dye stuffs
(4) All of the above

9. Two volatile and miscible liquids can be separated by fractional distillation into pure components under the conditions when :
(1) They have low boiling points.
(2) The difference in their boiling points is large.
(3) The boiling points of the liquids are close to each other.
(4) They do not form azeotropic mixture.

10. A fractional column is used in:
(1) Sublimation (2) Distillation
(3) Fractional distillation (4) Chromatography

11. Glycerol is purified by:
(1) Steam distillation (2) Vacuum distillation
(3) Fractional distillation (4) Simple distillation

12. Anthracene is purified by
(1) Filtration (2) Distillation
(3) Crystallisation (4) Sublimation

QUALITATIVE ANALYSIS

13. The compound that does not give a blue colour in Lassaigne's test is-
(1) Aniline (2) Glycine
(3) Hydrazine (4) Urea

14 In Lassaigne test thiourea is converted into-
(1) NaCNS (2) Na_2S
(3) NaCN (4) Na_2SO_4

15. In the Lassaigne's test, one of the organic compounds give red colour with $FeCl_3$. Compound can be:
(1) Na_2S (2) NH_2CSNH_2
(3) C_6H_5Cl (4) NaCN

16. Lassaigne's test is used in qualitative analysis to detect
(1) Nitrogen (2) Sulphur
(3) Chlorine (4) All of these

17. The compound that does not give a blue colour in Lassaigne's test is
(1) $C_6H_5—NH_2$ (2) CH_3CONH_2
(3) $NH_2—NH_2$ (4) $C_6H_5—NO_2$

18. Nitrogen containing organic compound when fused with sodium metal forms:
(1) $NaNO_2$ (2) NaCN
(3) $NaNH_2$ (4) NaNC

19. The sodium extract of an organic compound on acidification with acetic acid and addition of lead acetate solution gives a black precipitate. The organic compound contains
(1) Nitrogen (2) Halogen
(3) Sulphur (4) Phosphorus

20. The prussian blue colour obtained during the test of nitrogen by Lassaigne's test is due to the formation of:
(1) $Fe_4[Fe(CN)_6]_3$ (2) $Na_3[Fe(CN)_6]$
(3) $Fe(CN)_3$ (4) $Na_4(Fe(CN)_5NOS]$

21. Copper wire test of halogens is known as:
(1) Liebig's test (2) Lassaigne's test
(3) Fusion test (4) Beilstein's test

QUANTITATIVE ANALYSIS

22. If 0.32 gm of an organic compound containing sulphur produces 0.233g of $BaSO_4$ Then the percentage of sulphur in it is-
(1) 10 (2) 15
(3) 20 (4) 25

23. In Duma'smethod, the gas which is collected in Nitrometer is-
(1) N_2 (2) NO
(3) NH_3 (4) H_2

24. In Kjeldahl's method, $CuSO_4$ acts as-
(1) Oxidising agent (2) Catalytic agent
(3) Reducing agent (4) Hydrolysing agent

25. Nitrogen in an organic compound can be estimated by-
(1) Kjeldahl's method only (2) Duma's method only
(3) Both method (4) None of these

26. In Kjeldahl's method, nitrogen present in estimated as :
(1) N_2 (2) NH_3
(3) NO_2 (4) None of these

27. 6 g of the organic compound on heating with NaOH gave NH_3 which is neutralised by 200 mL of 1N HCl.
Percentage of nitrogen is:
(1) 12% (2) 60%
(3) 46.67% (4) 26.67%

28. The dessicants used for absorbing water during Liebig's method for estimation of carbon and hydrogen are:
(1) $CaCl_2$ (2) Na_2SO_4
(3) $MgSO_4.7H_2O$ (4) $CaHCO_3$

29. 0.28g of a nitrogenous compound was subjected to Kjeldahl's process to produce 0.17 g of NH_3. The percentage of nitrogen in the organic compound is :
(1) 5 (2) 20
(3) 50 (4) 80

PRABAL EXERCISE-2 (LEARNING PLUS)

1. 0.2 g of an organic compounds on complete combustion produces 0.44 g of CO_2, then the percentage of carbon in it is-
(1) 50 (2) 60
(3) 70 (4) 80

2. Which is useful for separating benzoic acid from a mixture of benzoic acid and methyl benzoate-
(1) Aq. $NaHCO_3$ (2) Dil. HCI
(3) Dil. H_2SO_4 (4) Dil. HNO_3

3. The process of differential extraction is based upon-
(1) Differential solubilities
(2) Differential molecular masses
(3) Different boiling points
(4) Different chemical properties

4. 0.2 g of an organic compound on complete combustion produces 0.18 g of water, then the percentage of hydrogen in it is-
(1) 5 (2) 10
(3) 15 (4) 20

5. In column chromatography the MOVING PHASE is constituted by-
(1) A substance which have to be separated
(2) Eluent
(3) Adsorbent
(4) Mixture of eluent and substances to be separated

6. Silica gel is used for keeping away the moisture because it-
(1) Absorbs H_2O (2) Adsorbs H_2O
(3) Reacts with H_2O (4) None of these

7. Which process is suitable for the purification of aniline-
(1) Simple distillation
(2) Steam distillation
(3) Fractional distillation
(4) Fractional crystallisation

8. Steam distillation is a better method of purification for_____ compounds-
(1) Liquids
(2) Steam volatile
(3) Non-volatile
(4) Miscible with water

9. In steam distillation the vapour pressure of the volatile organic compound is-
(1) Equal to atmospheric pressure.
(2) Less than the atmospheric pressure.
(3) More than the atmospheric pressure.
(4) Just double the atmospheric pressure.

10. Ellution is the process for-
(1) Crystallization of compound.
(2) Separation of compound.
(3) Extraction of compound.
(4) Distillation of compound.

11. Boiling point of a liquid can be increased by-
(1) Increasing the pressure.
(2) Decreasing the pressure.
(3) Purifying the liquid.
(4) Adding water to it.

12. In organic compounds P is estimated as-
(1) H_3PO_4 (2) P_2O_5
(3) $Mg_3(PO_4)_2$ (4) $(NH_4)_3\ PO_4\ .12MoO_3$

13. Which statement apply best to vacuum distillation-
(1) Distil liquids quickly with decomposition.
(2) It is very easy to distill.
(3) Distil liquid to avoid decomposition.
(4) None of these

14. A mixture of naphthalene and benzoic acid can be separated by-
(1) Extraction with solvent
(2) Sublimation
(3) Fractional crystallisation
(4) Distillation

15. If on adding $FeCl_3$ solution to acidified Lassaigne's solution, a blood red colouration is produced, it indicates the presence of -
(1) S (2) N
(3) N and S (4) S and Cl

16. In carius tube the compound $ClCH_2$—COOH was heated with fuming HNO_3 and $AgNO_3$.
After filteration and washing, a white ppt. was formed. The ppt is-
(1) AgCl (2) $AgNO_3$
(3) Ag_2SO_4 (4) $CH_2(ClCOOAg)_2$

17. During Lassaigne's test, N and S present in an organic compound changes into-
(1) Na_2S and NaCN (2) NaSCN
(3) Na_2SO_4 and NaCN (4) Na_2S and NaCNO

18. Two solids A and B have appreciable different solubilities in water but their melting points are very close. The mixture of A and B can be separated by-
(1) Sublimation
(2) Fractional crystallisation
(3) Distillation
(4) Specific method

19. The sodium extract prepared from sulphanilic acid, contains SCN^-. It gives blood red colouration with:
(1) a mixture of Na_2S and CS_2
(2) $FeCl_3$
(3) $FeSO_4$
(4) Na_2SO_3

20. Kjeldahl's method cannot be used for the estimation of nitrogen in:
(1) Pyridine (2) Nitrocompounds
(3) Azo compounds (4) All of these

21. The latest technique used for purification of organic compounds is:
(1) Chromatography (2) Vacuum distillation
(3) Fractional distillation (4) Crystallization

22. 0.765 g of an acid gives 0.535 g of CO_2 and 0.138 g of H_2O. Then the ratio of the percentage of carbon and hydrogen is:
(1) 19 : 2 (2) 18 : 11
(3) 20 : 17 (4) 1 : 7

23. 2.79 g of an organic compound when heated in Carius tube with conc. HNO_3 and H_3PO_4 formed converted into $MgNH_4$. PO_4 ppt. The ppt. on heating gave 1.332 g of $Mg_2P_2O_7$. The percentage of P in the compound is
(1) 23.33 (2) 13.33
(3) 33.33 (4) 26.66

24. In Lassaigne's solution, pink/violet colour is produced when sodium nitroprusside solution is added. It indicates the presence of :
(1) sulphur (2) nitrogen
(3) chlorine (4) none of these

25. The sodium extract of an organic compound on boiling with HNO_3 and addition of ammonium molybdate solution gives a yellow precipitate. The compound contains:
(1) Nitrogen (2) P
(3) S (4) Cl

26. The sodium extract of an organic compound on treatment with $FeSO_4$ solution, $FeCl_3$ and HCl gives a red solution. The organic compound contains:
(1) Both nitrogen and sulphur
(2) Nitrogen only
(3) Sulphur only
(4) Halogen

27. In Carius method of estimation of halogen, 0.15 g of an organic compound gave 0.12 g of AgBr. What is the percentage of bromine in the compound.
(1) 18% (2) 94%
(3) 63% (4) 34%

28. A miscible mixture of C_6H_6 + $CHCl_3$ can be separated by
(1) Sublimation (2) Distillation
(3) Filtration (4) Crystallization

29. Which of the following will not be soluble in sodium hydrogen carbonate ?
(1) 2, 4, 6-trinitrophenol
(2) Benzoic acid
(3) o-Nitrophenol
(4) Benzenesulphonic acid

30. Paper chromatography is an example of:
(1) Column chromatography
(2) Adsorption chromatography
(3) Partition chromatography
(4) Thin layer chromatography

31. Which is incorrect for paper chromatography
(1) It is a part of partition chromatography.
(2) It is a stationary phase.
(3) Rf value decrease when rate of adsorption increase.
(4) None of these

32. In Kjeldahls method of estimation of nitrogen, K_2SO_4 acts as
(1) catalytic agent (2) boiling point elevator
(3) hydrolysis agent (4) oxidizing agent

33. During formation of sodium extract, generally the substance that ignites is
(1) Na (2) O_2
(3) CO_2 (4) H_2

34. Sorbic acid consists of C = 64.3%. H = 7.2% and 28.5% oxygen. Find the empirical formula.
(1) $C_2H_4O_3$ (2) $C_3H_4O_2$
(3) C_3H_4O (4) CH_2O

35. A mixture of benzene and aniline can be separated by
(1) alcohol
(2) NaOH
(3) HCl
(4) Hot water

PARIKSHIT EXERCISE-3 (MULTICONCEPT)

MATCH THE COLUMN TYPE QUESTIONS

1. Match the following

	Method used for detections		**Element detections**
	Column-I		**Column-II**
A.	Liebig's method	P.	Carbon
B.	Duma's method	Q.	Nitrogen
C.	Carius method	R.	Sulphur
D.	Kjeldahl's method	S.	Hydrogen
		T.	Halogen

(1) A–(P,S); B–(Q); C–(R,T); D–(Q)
(2) A–(P,Q); B–(S); C–(R,S); D–(Q)
(3) A–(P,R); B–(R); C–(Q,T); D–(P)
(4) A–(Q,S); B–(P); C–(S,T); D–(S)

2. Match the following

	Column-I		**Column-II**
	Element		**Coloured ppt formed**
A.	Nitrogen	P.	Black sulphide
B.	Sulphur	Q.	Violet Complex
C.	Chloride	R.	Prussian Blue
D.	Bromide	S.	Light yellow
		T.	White

(1) A–(R); B–(P); C–(T); D–(S)
(2) A–(P); B–(S); C–(P); D–(Q)
(3) A–(P); B–(R); C–(Q); D–(P)
(4) A–(Q); B–(P); C–(R); D–(S)

3. Match the compound in Column-I to the best technique of separation listed in Column-II.

	Column-I		**Column-II**
A.	Camphour containing non-volatile impurities	P.	Differential extraction
B.	Impure sample of aniline	Q.	Sublimation
C.	Ortho and para nitro-anilines	R.	Distillation
D.	Ether from ethyl alcohol	S.	Chromatography

(1) A–(Q); B–(R); C–(R,S); D–(R)
(2) A–(R); B–(R); C–(R,P); D–(Q)
(3) A–(P); B–(Q); C–(R,S); D–(S)
(4) A–(R); B–(Q); C–(S,P); D–(S)

4. The correct match between Column-I and Column-II is:

	Column-I		**Column-II**
(A)	Benzaldehyde	P.	Mobile phase
(B)	Alumina	Q.	Adsorbent
(C)	Acetonitrile	R.	Adsorbate

(1) (A)–(Q); (B)–(P); (C)–(R)
(2) (A)–(R); (B)–(Q); (C)–(P)
(3) (A)–(Q); (B)–(R) ; (C)–(P)
(4) (A)–(P); (B)–(R); (C)–(Q)

5. The correct match between Column-I and Column-II is:

	Column-I (Mixture)		Column-I (Separation method)
A.	H_2O: Sugar	P.	Sublimation
B.	H_2O: Aniline	Q.	Recrystallization
C.	H_2O: Toluene	R.	Steam distillation
		S.	Differential extraction

(1) (A)–(S); (B)–(R); (C)–(P)
(2) (A)–(Q); (B)–(R); (C)–(S)
(3) (A)–(R); (B)–(P); (C)–(S)
(4) (A)–(Q); (B)–(R); (C)–(P)

6. Match Column-I with Column-II:

	Column-I (Test/Reagent/ Observation)		Column-II (Species detected)
A.	Lassaigne's Test	P.	Carbon
B.	Cu (II) oxide	Q.	Sulphur
C.	Silver nitrate	R.	N, S, P, and halogen
D.	The sodium fusion extract gives black precipitate with acetic acid and lead acetate.	S.	Halogen Specifically

The correct match is:
(1) A–(P); B–(Q); C–(R); D–(S)
(2) A–(P); B–(R); C–(S); D–(Q)
(3) A–(R); B–(P); C–(S); D–(Q)
(4) A–(S); B–(R); C–(Q); D–(P)

7. Match Column-I with Column-II:

	Column-I (Mixture)		Column-II (Purification Process)
A.	Chloroform and Aniline	P.	Steam distillation
B.	Benzoic acid and Naphthalene	Q.	Sublimation
C.	Water and Aniline	R.	Distillation
D.	Naphthalene & Sodium chloride	S.	Crystallisation

(1) (A)–(S), (B)–(R), (C)–(P), (D)–(Q)
(2) (A)–(R), (B)–(P), (C)–(S), (D)–(Q)
(3) (A)–(R), (B)–(S), (C)–(Q), (D)–(P)
(4) (A)–(R), (B)–(S), (C)–(P), (D)–(Q)

8. Match the type of mixture of compounds in Column I with the technique of separation/purification given in column II.

	Column I		Column II
A.	Two solids which have different solubilities in a solvent and which do not undergo a reaction when dissolved in it	P.	Steam distillation
B.	Liquid that decomposes at its boiling point	Q.	Fractional distillation
C.	Steam volatile liquid	R.	Crystallisation
D.	Two liquids that have boiling points close to each other	S.	Distillation under reduced pressure

(1) (A)-(S), (B)-(R), (C)-(P), (D)-(Q)
(2) (A)-(R), (B)-(P), (C)-(S), (D)-(Q)
(3) (A)-(R), (B)-(S), (C)-(Q), (D)-(P)
(4) (A)-(R), (B)-(S), (C)-(P), (D)-(Q)

Correct-Incorrect Statement Mcqs

9. Which of the following is incorrect statement?
(1) Carius tube used in the estimation of sulphur in an organic compound.
(2) Kjeldahl's method is used for the estimation of nitrogen in an organic compound.
(3) Phosphoric acid produced on oxidation of phosphorus present in an organic compound is precipitated as Mg2P2O7 by adding magnesia mixture.
(4) Carius method is used for the estimation of nitrogen in an organic compound.

10. In chromatography, which of the following statement is INCORRECT for Rf?
(1) Rf value depends on the type of chromatography.
(2) The value of Rf cannot be more than one.
(3) Higher Rf value means higher adsorption.
(4) Rf value is dependent on the mobile phase

11. If dichloromethane (DCM) and water (H2O) are used for differential extraction, which one of the following statements is correct?
(1) DCM and H2O would stay as lower and upper layer respectively in the S.F.
(2) DCM and H2O will make turbid/colloidal mixture
(3) DCM and H2O would stay as upper and lower layer respectively in the separating funnel (S.F.)
(4) DCM and H2O will be miscible clearly.

12. The correct statement for Lassaigne's test regarding sodium extract:
(1) It is used to form sodium derivative which are non-soluble.
(2) It is used to convert elements into soluble ionic compound.
(3) It is used to make element flammable.
(4) It is used as an oxidising agent.

13. Incorrect statement about chromatography:
(1) It can be used to separate ortho and para nitro-anilines.
(2) It is used to separate blue and red dyes.
(3) It is used to purify plant pigments.

(4) It is used to separate compounds having dissimilar boiling points.

14. Incorrect statement regarding differential extraction:

(1) Organic compound should be in aqueous medium.

(2) Organic solvent is used which is immiscible in water.

(3) Organic compound is more soluble in organic solvent than water.

(4) Very small quantity of solvent is required to extract small quantity of compound.

15. Which of the following statements is incorrect?

(1) In the estimation of carbon, an organic compound is heated with CaO in a combustion tube.

(2) Using Lassaigne's test nitrogen and sulphur present in an organic compound can be tested.

(3) Using Beilstein's test the presence of halogen in a compound can be tested.

(4) Lassaigne's tests fails to identify nitrogen in diazo compound.

STATEMENT BASED MCQS

(1) Both Statement-I and Statement-II are correct.

(2) Both Statement-I and Statement-II are incorrect.

(3) Statement-I is correct and Statement-II is incorrect.

(4) Statement-I is incorrect and Statement-II is correct.

16. **Statement-I:** In 'Lassaigne's Test, when both nitrogen and sulphur are present in an organic compound, sodium thiocyanate is formed.

Statement-II: If both nitrogen and sulphur are present in an organic compound, then the excess of sodium used in sodium fusion will decompose the sodium thiocyanate formed to give NaCN and Na2S.

17. **Statement-I:** Retardation factor (Rf) can be measured in meter/centimetres.

Statement-II: Rf value of a compound remains constant in all solvents.

18. **Statement-I:** A mixture of chloroform and aniline can be separated by simple distillation.

Statement-II: When separating aniline from a mixture of aniline and water by steam distillation aniline boils below its boiling point.

ASSERTION & REASON MCQS

(1) If both Assertion (A) and Reason (R) are True and the Reason (R) is a correct explanation of the Assertion (A).

(2) If both Assertion (A) and Reason (R) are True but Reason (R) is not a correct explanation of the Assertion (A).

(3) If Assertion (A) is True but the Reason (R) is False.

(4) Assertion (A) is False but Reason (R) is True.

19. **Assertion (A):** Thin layer chromatography is an adsorption chromatography.

Reason (R): A thin layer of silica gel is spread over a glass plate of suitable size in thin layer chromatography which acts as an adsorbent.

20. **Assertion (A):** A mixture contains benzoic acid and naphthalene. The pure benzoic acid can be separated out by the use of benzene.

Reason (R): Benzoic acid is soluble in hot water.

21. **Assertion:** To detect presence of halogens in an organic compound, some dilute nitric acid is added.

Reason: Dilute HNO_3 destroys Na_2S and NaCN by oxidation.

ANSWER KEY

CONCEPT APPLICATION

1. (2) **2.** (1) **3.** (4) **4.** (4) **5.** (3) **6.** (2) **7.** (1) **8.** (2) **9.** (4) **10.** (*a*)

SCHOOL LEVEL PROBLEMS

Single choice type questions

1. (3) **2.** (4) **3.** (2) **4.** (4) **5.** (4) **6.** (2) **7.** (3) **8.** (4)

Assertion and reason type questions

1. (1) **2.** (3)

Match the Column type Questions

1. (1) **2.** (1)

Case Based Study Questions

1. (3) **2.** (4) **3.** (3) **4.** (4)

PRARAMBH EXERCISE-1 (TOPICWISE)

1. (4) **2.** (3) **3.** (3) **4.** (4) **5.** (2) **6.** (4) **7.** (2) **8.** (4) **9.** (4) **10.** (3)
11. (2) **12.** (4) **13.** (3) **14.** (1) **15.** (2) **16.** (4) **17.** (3) **18.** (2) **19.** (3) **20.** (1)
21. (4) **22.** (1) **23.** (1) **24.** (2) **25.** (3) **26.** (2) **27.** (3) **28.** (1) **29.** (*c*)

PRABAL EXERCISE-2 (LEARNING PLUS)

1. (2) **2.** (1) **3.** (1) **4.** (2) **5.** (4) **6.** (2) **7.** (2) **8.** (2) **9.** (2) **10.** (2)
11. (1) **12.** (4) **13.** (3) **14.** (2) **15.** (3) **16.** (1) **17.** (1) **18.** (2) **19.** (2) **20.** (4)
21. (1) **22.** (1) **23.** (2) **24.** (1) **25.** (2) **26.** (1) **27.** (4) **28.** (2) **29.** (3) **30.** (3)
31. (2) **32.** (2) **33.** (4) **34.** (3) **35.** (3)

PARIKSHIT EXERCISE-3 (MULTICONCEPT)

1. (1) **2.** (1) **3.** (1) **4.** (2) **5.** (2) **6.** (3) **7.** (4) **8.** (4) **9.** (4) **10.** (3)
11. (1) **12.** (2) **13.** (4) **14.** (4) **15.** (1) **16.** (1) **17.** (2) **18.** (1) **19.** (1) **20.** (4)
21. (3)