

PURIFICATION & CHARACTERISATION OF ORGANIC COMPOUNDS**METHODS OF PURIFICATION OF ORGANIC COMPOUNDS :**

Organic compounds obtained either from natural source (or) synthesized in laboratory 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) Crystallisation |
| (3) Distillation | (4) Solvent extraction (differential extraction) |
| (5) Chromatography. | |

Note: Most of the pure compounds contain sharp Melting point & Boiling points.

Sublimation :

The process of conversion of sublimable solid to vapour state directly by heating without passing through liquid state is called sublimation.

Solid vapour

- This method is used for purification of solids
- Sublimation process is used for separation of sublimable volatile compounds from non sublimable impurities.
- Sublimation is generally used for purification of camphor, naphthalene. Anthracene. Benzoic acid, phthalic anhydride, Anthraquinone, Indigo and Iodine HgCl_2 , solid SO_3

Crystallisation :

- It is used for purification of solid organic compounds.
- Crystallisation is based on the difference in solubilities of the compound and impurities in a suitable solvent.
- The principle involved in this method is impure compound dissolved in a solvent is sparingly soluble at low temperature, but appreciably soluble at high temperature. Insoluble impurities are separated by filtration. The filtrate on cooling saturated solution, pure compound crystallises out. If a compound is highly soluble in one solvent and too little soluble in another solvent then crystallisation is carried out by using mixture of these solvents.
- Impurities, which impart colour are removed by adsorbing over activated charcoal.
- Repeated crystallisation is required if organic compound contains impurities of comparable solubilities.
- The process of separation of different components of a mixture by repeated crystallisation is called fractional crystallisation.
- Fractional crystallisation is used for separation of two or more soluble substances which have different solubilities in the same solvent.
- Most commonly used solvents for crystallisation are water, alcohol, ether, chloroform, carbontetrachloride. acetone, benzene, petroleum ether.

- Sugar having an impurity of common salt can be crystallised from hot ethanol. since sugar dissolves in hot ethanol but common salt does not.
- Fractional crystallisation can be used to separate a mixture of KClO_3 (less soluble) and KCl (more soluble).

DISTILLATION :

Distillation is an important method used to separate

- i) Volatile liquids from non volatile impurities.
- ii) Liquids having sufficient difference in boiling points.

Simple Distillation :

- This process is used for purification of liquids which does not undergo decomposition at their boiling points
- The vapourisation of a liquid by heating and subsequent condensation of vapours by cooling is known as distillation.
- Liquid mixture is taken in a round bottom flask and heated carefully, the vapour component with lower boiling point distills first, the vapour formed is condensed by using condenser and the liquid is collected in a receiver. The vapours of component with higher boiling point distills later.
- The liquids that have boiling point difference greater than 40°C can be purified by this method,

e.g:

- (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. 457 K)

Fractional Distillation :

- Fractional distillation is used if the difference in boiling point of two liquids is less than 40°C .
- Vapours of liquid mixture are passed through fractionating column before condensation, which is fitted over mouth of the round bottom flask.
- Vapours of liquid with higher boiling point condense before the vapours of liquid with lower boiling point the vapours raising up in the fractionating column is richer in more volatile component.
- Fractionating column provides many surfaces for heat exchange between ascending vapours and descending condensed liquid.
- Each successive condensation and vapourisation unit in the fractionating column is called a theoretical plate.
- Liquids forming a constant boiling mixture (azeotropic mixture) can not be separated by this method.
- Fractional distillation is used to separate different fractions of crude oil in petroleum industry.
- This method is used for separation of mixture of acetone (B.P. 330K) and methyl alcohol (B.P. 338K)
- Mixture of benzene and toluene can be separated by fractional distillation.

Ex.1 How is ethyl alcohol purified from methylated spirit.

Sol: Methylated spirit is ethyl alcohol contaminated mainly with methyl alcohol. Ethyl alcohol is purified by fractional distillation since the difference in boiling point is less.

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.
- These liquids are made to boil at a temperature lower than their normal boiling point by reducing pressure on their surface with the help of vacuum pump.
- Glycerine, H_2O_2 , formaldehyde are purified by vacuum distillation.
- Glycerol can be separated from spent-lye in soap industry by using vacuum distillation.
- Sugar cane 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
 - i) are steam volatile
 - ii) are insoluble in water.
 - iii) Process high vapour pressure (10-15 mm of Hg at 373 K)
 - iv) Contains non volatile impurities.
- Steam distillation is based on Dalton's law of partial pressure i.e., $P = P_1 + P_2$. where P = Atmospheric pressure.
 - P_1 = Partial pressure of organic liquid
 - P_2 = Vapour pressure due to water.
- Compounds which can be purified by steam distillation are aniline, nitrobenzene, bromobenzene, o-nitrophenol, o-hydroxy benzaldehyde (salicylaldehyde), o-hydroxy acetophenone, turpentine oil, essential oils.

Ex.2 Mention about the purification of (a) aniline and (b) naphthalene

- Sol.** (a) Aniline can be purified by steam distillation because it is immiscible with water and steam volatile.
(b) Naphthalene can be purified by sublimation because it changes on heating directly to vapour state and on cooling, it changes back into solid form.

Solvent Extraction (Differential Extraction) :

- **Definition:** 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 separator funnel).
- Organic solvent is distilled or evaporated to get organic compound.
- If organic compound is less soluble in organic solvent then 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.

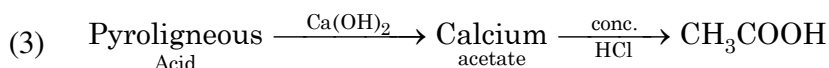
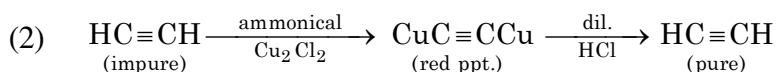
- Ether is a better solvent in differential extraction due to:
 - i) its less polarity ii) less reactivity
 - iii) Less Boiling Point iv) higher solubility of organic compounds

SEPARATION BY CHEMICAL METHODS :

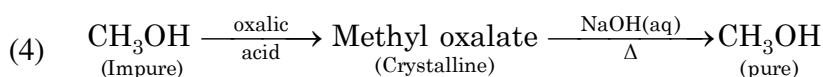
- It is used for mixture of substances which are chemically different.

e.g :

- (1) Separation of acidic and basic compounds of coal-tar.



(from wood distillation industry) $\rightarrow \text{CH}_3\text{COOH} + \text{MeCoMe} + \text{MeOH}$



Chromatography :

- This method is used for separation of mixtures into their components, purification of compounds and also to test the purity of compounds.
- Chromatography is obtained from the greek word "Chroma" means colour and "graphy" means writing.
- This method was first used for separation of coloured substances found in plants.
- This method was described by Tswett.
- This Technique consists of two phases one is stationary phase of large surface area while the second is moving phase which is allowed to move slowly over the stationary phase.
- Stationary phase is either liquid or solid, while moving phase may be liquid or gas.
- The technique of chromatography is based on the rates at which the components of the mixture moves through a porous medium (called stationary phase) under the influence of some solvent (or) gas (called mobile phase).
- Mixture of substances is applied on a stationary phase which may be solid or liquid. A pure solvent, a mixture of solvents or a gas is allowed to move slowly over the stationary phase, the components of the mixture get gradually separated from one another.
- Recovery of separated substances by using suitable solvent is known as elution. The solvent used is known as eluant.

S.No.	Chromatography process	Stationary Phase	Mobile Phase
1.	Column chromatography (Adsorption)	Solid	Liquid
2.	Liquid-liquid partition chromatography	Liquid	Liquid
3.	Paper chromatography	Liquid	Liquid
4.	Thin layer chromatography (TLC)	Liquid (or) solid	Liquid
5.	Gas-liquid chromatography (GLC)	Liquid	Gas
6.	Gas-solid chromatography (GSC)	Solid	Gas
7.	Ionic exchange chromatography	Solid	Liquid

- Based on the principle involved chromatography is classified in to
 - Adsorption chromatography and
 - partition chromatography.
- (a) **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, alumina, magnesium oxide, cellulose powder, activated animal charcoal.
 - When a mobile phase is allowed to move over stationary phase, the components of the mixture move by varying distances over stationary phase.
 - There are two main types of chromatographic techniques based on principle of differential adsorption
 - Column chromatography and
 - Thin layer chromatography (TLC)

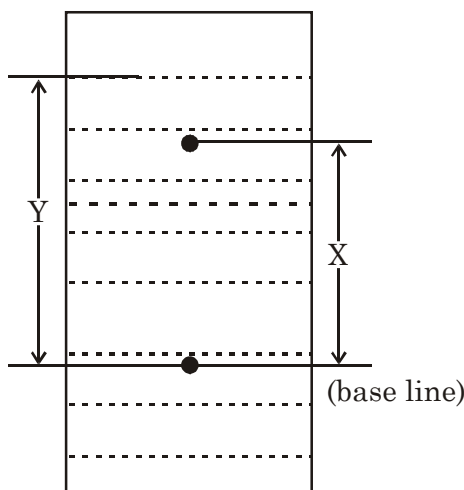
i) Column Chromatography

- It involves separation of a mixture over a column of adsorbent packed in a glass tube, which is fitted with stop cock at its lower end.
- The mixture to be separated on the adsorbent is placed at the top of the stationary phase.
- An appropriate eluant, which is a liquid or a mixture of liquids is allowed to flow down the column slowly.
- The strongly adsorbed substances are retained near the top and others come down to various distances in the column,

(ii) Thin layer chromatography (TLC)

- It involves separation of substances of a mixture over a thin layer of an adsorbent coated on glass plate.
- The glass plate is coated with adsorbent (ex: silica gel, alumina) as a thin layer (about 0.2mm thick) is called chromatography plate or chroma plate .
- The solution of mixture to be separated is applied as small spot about 2cm above from one end of the TLC plate.

- The glass plate is placed in a closed jar containing the eluant. As the eluant rises up, the components of the mixture move up along 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.



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

- The spots of coloured compounds are visible on TLC plate due to their original colour.
 - The colourless compound which fluoresce are detected with ultraviolet light
 - Spots of compounds are even detected by allowing them to adsorb iodine, will show up as brown spots.
 - Some times an appropriate reagent is sprayed on the plate.
- eg :** Amino acids are 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 known as chromatography paper is used.
- In Chromatography paper, cellulose helps as support, and water absorbed from air on to hydroxyl groups of cellulose acts as stationary phase.
- The chromatography paper spotted with the solution of mixture at the base is suspended in a suitable solvent or mixture of solvents, this solvent (s) act as mobile phase.
- The solvent rises up the paper by capillary action and flows over the spot.
- The paper selectively retains different component according to their differing partition in the two phases. The paper strip so developed is called 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 compound may be observed either under ultraviolet or by the use of appropriate spraying agent

Additional Information : Applications of Chromatography:

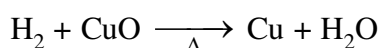
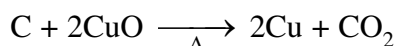
- I. i) In Chemical Industry column chromatography is used for separation of required components obtained after synthesis.
ii) TLC is useful for monitoring large scale column chromatography.
- II **Pharmaceutical industry** : Chromatography is used for separation of chiral compounds to obtain pharmaceutically active optical isomer.
- III **Food Industry** : Chromatography techniques are used for quality control in food industry. It is used to determine presence and to separate additives, flavours etc. It is also used to detect presence of contaminants like mould, bacteria in food.
- IV **Environment-Testing lab** : Presence and quality of pollutants in air and drinking water can be determined by chromatography technique.
- V) **Diagnostic Technique**: Presence of certain drugs and the marker compounds for medical diagnosis in blood and urine are determined.

Qualitative Analysis of Organic Compounds (Detection of Elements):

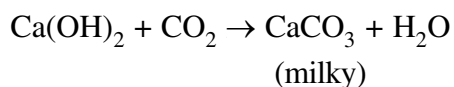
- The qualitative analysis of an organic compound involves detection of all elements present in it

Detection of Carbon and Hydrogen :

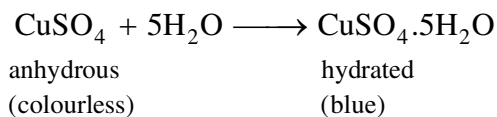
- Carbon and hydrogen are detected by heating the compound with cupric oxide (CuO).
- Carbon present in the compound is oxidised to carbon dioxide, which turns lime water milky.
- Hydrogen present in the compound is converted into water, which turns anhydrous copper sulphate into blue.



- Carbondioxide turns lime water milky.



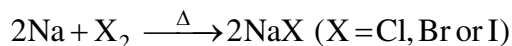
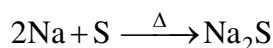
- Water vapours turn anhydrous copper sulphate into blue.



Detection of Nitrogen, Sulphur Halogens & Phosphorus :

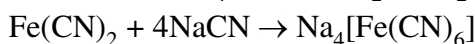
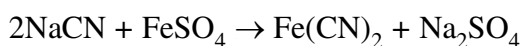
- Nitrogen, sulphur, halogens and phosphorus present in an organic compound are detected by Lassaigne's test.
- Organic compounds are fused with dry sodium in fusion tube and fused mass after extraction with water is boiled and filtered, the filtrate is called sodium fusion extract
- During preparation of sodium fusion extract covalent compound is converted into ionic compound.

- The following reactions takes place



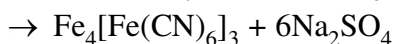
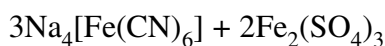
Test for Nitrogen :

Sodium fusion extract is boiled with freshly prepared ferrous sulphate (FeSO_4) solution and then acidified with concentrated sulphuric acid. The formation of Prussian blue colour confirms the presence of nitrogen.



sodium hexacyanoferrate(II)

On heating with concentrated sulphuric acid some Iron(II) ions are oxidised to Iron(III) ion, which reacts with sodium hexacyanoferrate(II) to produce Iron (III) hexacyanoferrate(II) (ferri ferrocyanide) which is Prussian blue in colour.



ferriferrocyanide

(prussianblue)

- This test fails in case of diazo compounds.
- If the amount of nitrogen present is less, then Prussian blue is present in collaidal form and the solution looks green.

Ex.3 Hydrazine does not give Lassaigne's test, Why?

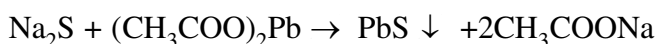
Sol. In the Lassaigne's test, nitrogen is converted to cyanide by combining with carbon of the compound. Hydrazine does not contain carbon and hence cyanide cannot form.

Ex.4 Why diazonium salts do not show positive Lassaigne's test for nitrogen ?

Sol: 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.

Test for Sulphur:

- Sodium fusion extract is acidified with acetic acid and lead acetate is added to it, a black precipitate of lead sulphide is formed, which indicates presence of sulphur.



black

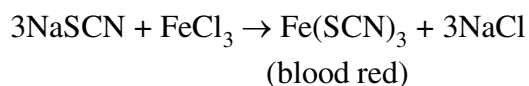
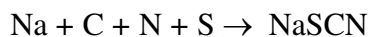
- Sodium fusion extract is treated with freshly prepared sodium nitroprusside, appearance of violet colour (purple) indicates presence of sulphur.



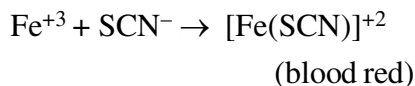
(sodium nitro prusside)

(violet)

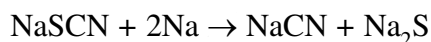
- In case both nitrogen and sulphur are present in an organic compound sodium thiocyanate is formed, which gives blood red colour with neutral FeCl_3 solution.



(or)



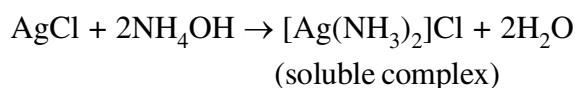
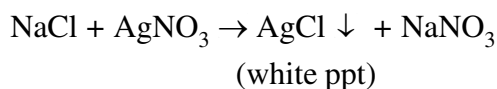
- If sodium fusion is carried out with excess of sodium, the thiocyanate decomposes to yield cyanide and sulphide, these ions gives their usual tests.



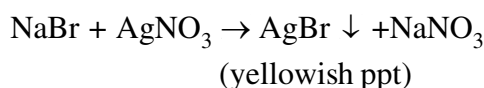
Test for Halogens:

- Sodium fusion extract is acidified with nitric acid and then treated with silver nitrate solution.

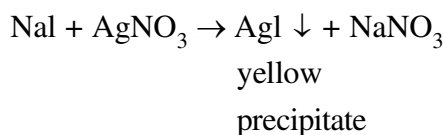
(i) White precipitate, soluble in ammonium hydroxide indicates presence of chlorine.



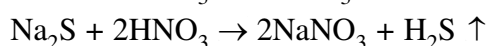
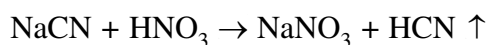
(ii) Yellowish precipitate, sparingly soluble in ammonium hydroxide indicates presence of bromine.



(iii) Yellow precipitate, insoluble in ammonium hydroxide indicates presence of Iodine.



- Nitrogen and sulphur are also present in the compound, the sodium fusion extract is boiled with concentrated nitric acid to decompose sodium cyanide & sodium sulphide formed during Lassaigne's test, otherwise they interfere with silver nitrate test for halogens.



- If NaCN and Na₂S are not decomposed, then white and black precipitates of AgCN and Ag₂S are formed respectively with silver nitrate solution.

Beilstein's Test:

A copper wire flattened at one end is heated in an oxidising flame of Bunsen burner. The heating is continued till it does not impart blue colour flame. The hot end of copper wire is now touched with the organic substance and is once again kept in flame, the appearance of green or blue colour indicates the presence of halogens in the organic compound.

Limitations :

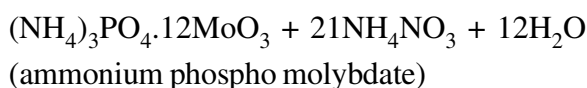
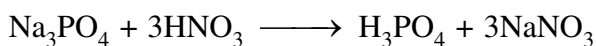
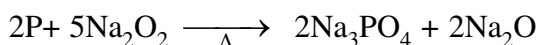
- Substances such as urea, thiourea do not contain halogens but gives this test
- It does not tell which halogen is present in organic compound.

Chlorine Water Test for Bromine and Iodine :

- Both AgBr and AgI are yellow precipitates, it is a little bit difficult to identify given halogen in bromine or iodine, to confirm it chlorine water test is used.
- Sodium fusion extract is acidified with dilute H_2SO_4 (or) HNO_3 , to this 1(or) 2ml of chloroform (or) Carbon tetrachloride is added and then excess of chlorine water is added with constant shaking.
- (i) If chloroform (or) carbon tetrachloride layer becomes yellow (or) brown indicates presence of bromine.
 $2\text{NaBr} + \text{Cl}_2 \rightarrow 2\text{NaCl} + \text{Br}_2$
 Br_2 dissolves in chloroform (or) carbontetrachloride gives yellow (or) brown colour
- (ii) If chloroform (or) carbon tetrachloride layer becomes violet indicates presence of iodine
 $2\text{NaI} + \text{Cl}_2 \rightarrow 2\text{NaCl} + \text{I}_2$
 I_2 dissolves in chloroform (or) carbontetrachloride gives violet colour.
- Presence of NaCN (or) Na_2S in sodium fusion extract does not interfere in this test.

Test for Phosphorus :

The compound is heated with oxidising agent (sodium peroxide) or with fusion mixture (sodium carbonate and potassium nitrate) phosphorus present in the compound is oxidised to sodium phosphate. The solution is boiled with nitric acid and then treated with ammonium molybdate, a canary yellow (ammonium phospho molybdate) precipitate formation (or) yellow colouration indicates presence of phosphorus.

**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.

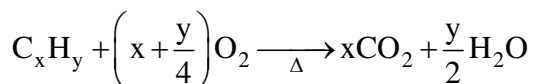
eg: $-\text{OH}$, $-\text{COOH}$, $-\text{CHO}$, $-\text{NO}_2$ etc

QUANTITATIVE ANALYSIS

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



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

$$\%C = \frac{12}{44} \times \frac{\text{weight of CO}_2 \text{ formed}}{\text{weight of organic compound}} \times 100$$

$$\%H = \frac{2}{18} \times \frac{\text{weight of CO}_2 \text{ formed}}{\text{weight of organic compound}} \times 100$$

Ex.5 On complete combustion, 0.246g of an organic compound gave 0.198 g of carbon dioxide and 0.1014g of water. Determine the percentage composition of carbon and hydrogen in the compound.

Sol. $\% \text{ of carbon} = \frac{12 \times 0.198 \times 100}{44 \times 0.246} = 21.95$

$$\% \text{ of hydrogen} = \frac{2 \times 0.1014 \times 100}{18 \times 0.246} = 4.58$$

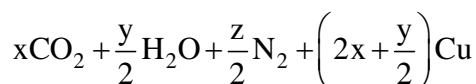
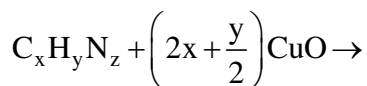
ESTIMATION OF NITROGEN

Nitrogen present in organic compound is estimated by

- (a) Dumas method
(b) Kjeldahl's method

(a) Dumas method

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



- Oxides of nitrogen + Cu \rightarrow N₂ + CuO
- The mixture of gases produced is collected over caustic potash solution (KOH solution) which absorbs CO₂.
- Nitrogen is collected in the upper part of nitro meter.

$$\% \text{N} = \frac{28}{22400} \times \frac{\text{Volume of nitrogen in ml at STP}}{\text{Weight of organic compound}} \times 100$$

Ex.6. 0.25g of an organic compound gave 30 cm³ 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 cm³

Temperature = 288 K

Pressure = 745 – 12.7 = 732.3 mm

Volume of dinitrogen at STP :

$$V_2 = \frac{P_1 V_1}{T_1} \times \frac{T_2}{P_2} = \frac{732.3 \times 30 \times 273}{288 \times 760} = 27.4 \text{ cm}^3$$

$$\text{Percentage of nitrogen in organic compound} = \frac{28}{22400} \times \frac{\text{vol of N}_2 \text{ at STP}}{\text{wt of organic compound}} \times 100$$

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

Ex.7 0.3g of an organic compound gave 50 ml nitrogen at 27°C and 715 mm pressure. If the aqueous tension at 27°C is 15 mm, calculate the percentage composition of nitrogen in the compound.

Sol. Mass of the substance = 0.3 g

Volume of the moist dinitrogen = 50 ml

Temperature = 27°C = 27 + 273 = 300 K

Pressure = 715 – 15 = 700 mm

Volume of dinitrogen at STP :

$$v_2 = \frac{P_1 V_1}{T_1} = \frac{T_2}{P_2} = \frac{700 \times 50 \times 273}{760 \times 300} = 41.9 \text{ ml}$$

$$\frac{28}{22400} \times \frac{\text{vol of N}_2 \text{ STP}}{\text{wt of organic compound}} \times 100 = \frac{28}{22400} \times \frac{41.9}{0.3} \times 100 = 17.46$$

22400 ml of dinitrogen at STP weight = 28 g

$$41.9 \text{ ml dinitrogen at STP weight} = \frac{28 \times 41.9}{22400} \text{ g}$$

$$0.3 \text{ g of organic compound contains } \frac{28 \times 41.9}{22400} \text{ g of N}_2$$

$$100 \text{ g of organic compound contain } \frac{28 \times 41.9}{22400} \times \frac{100}{0.3} = 17.46$$

percentage of nitrogen = 17.46

Kjeldahl's Method :

- In this method nitrogen 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. K_2SO_4 increases boiling point of H_2SO_4 and CuSO_4 acts as catalyst.
- Organic compound + $\text{H}_2\text{SO}_4 \longrightarrow (\text{NH}_4)_2\text{SO}_4$
- The resulting solution is distilled with excess of sodium hydroxide
 $(\text{NH}_4)_2\text{SO}_4 + 2\text{NaOH} \longrightarrow \text{Na}_2\text{SO}_4 + 2\text{NH}_3 + 2\text{H}_2\text{O}$
- 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.

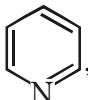
$$\text{Percentage of Nitrogen} = \frac{14}{1000} \times \frac{V \times N}{\text{wt. of organic compound}} \times 100$$

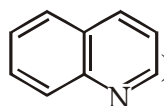
$$\%N = \frac{1.4 \times V \times N}{\text{Weight of organic compound}}$$

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 ($-\text{NO}_2$), Nitroso (NO), azo group

($-\text{N}=\text{N}-$), azoxy compounds $\left[\begin{array}{c} \text{O} \\ \uparrow \\ -\text{N} = \text{N}- \end{array} \right]$ and nitrogen present in the ring (pyridine , quinoline



because nitrogen present in these compounds is not quantitatively converted in to ammonium sulphate.

Ex.8 In Kjeldahl's estimation of nitrogen, the ammonia evolved from 0.5 g of an organic compound neutralised 10 ml of 1M H_2SO_4 . Calculate the percentage of nitrogen in the compound?

Sol. 10 ml of 1M $\text{H}_2\text{SO}_4 = 20\text{ml}$ of 1M NH_3
 1000 ml of 1M ammonia contains 14 g nitrogen

$$20 \text{ ml of 1M ammonia contains } \frac{14 \times 20}{1000} \text{ g N}_2$$

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

Ex.9 In Kjeldahl's estimation of nitrogen, the ammonia obtained from 0.5 g of an organic substance was passed

into 100 cm³ of $\frac{M}{10}$ NaOH for neutralisation. Calculate the percentage of nitrogen in the compound.

Sol. m eq. of H₂SO₄ taken = molarity × basicity × volume (ml)

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

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

$$\text{m. eq of H}_2\text{SO}_4 \text{ unused} = 20 - 15.4 = 4.6$$

$$\% \text{ of nitrogen} = \frac{1.4 \times \text{meq of H}_2\text{SO}_4 \text{ unused}}{\text{wt of organic compound}} = \frac{1.4 \times 4.6}{0.5} = 12.88\%$$

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 CO₂ and H₂O.
- Halogen present in the organic compound is converted into silver halide.
- The precipitate is washed, dried and weighed

$$\text{Percentage of halogen} = \frac{\text{Atomic weight of halogen}}{\text{Mwt of silver halide}} \times \frac{\text{Weight of silver halide formed}}{\text{Weight of organic compound}} \times 100$$

$$\% \text{Cl} = \frac{35.5}{143.5} \times \frac{\text{wt of AgCl formed}}{\text{wt of organic compound}} \times 100$$

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

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

Ex.10 In Carius method, 0.1890g of an organic compound gave 0.2870g of silver chloride. Calculate the percentage of chlorine in the compound

Sol: Weight of substance = 0.1890g

Weight of silver chloride = 0.2870g

$$\% \text{ of chlorine} = \frac{\text{weight of AgCl} \times 35.5 \times 100}{\text{weight of substance} \times 143.5} = \frac{0.2870 \times 35.5 \times 100}{0.1890 \times 143.5} = 37.8$$

Ex.11 One gram of bromoalkane on heating with excess silver nitrate in Carius tube method gave 0.94g of yellow precipitate. What is the percent weight of halogen ?

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

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

Ex.12 In carius method of estimation of halogen 0.15g of an organic compound gave 0.12g of AgBr. Find out the percentage of bromine in the compound

Sol. $\% \text{ of bromine} = \frac{80 \times 0.12 \times 100}{188 \times 0.15} = 34.04$

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 BaCl₂ solution gives precipitate of BaSO₄.
- It is filtered, the precipitate is washed, dried and weighed.

$$\%S = \frac{\text{Atomic wt of sulphur}}{\text{Mol. wt of BaSO}_4} \times \frac{\text{wt of BaSO}_4 \text{ formed}}{\text{wt of organic compound}} \times 100$$

$$\%S = \frac{32}{233} \times \frac{\text{wt of BaSO}_4 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

Ex.13 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 BaSO₄ contain 32g sulphur

$$0.4813 \text{ g BaSO}_4 \text{ contain } \frac{32 \times 0.4813}{233} \text{ g sulphur}$$

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

Ex.14 On heating 0.2g of an organic compound with a mixture of barium chloride and nitric acid, 0.466g of barium sulphate was obtained. Calculate the percentage of sulphur.

Sol. Weight of substance = 0.2 g

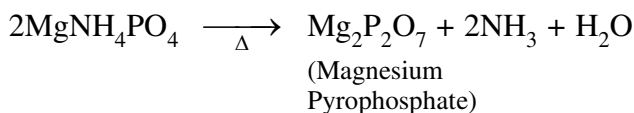
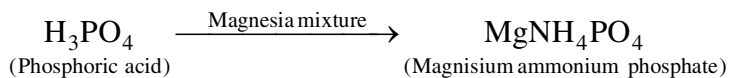
Weight of barium sulphate = 0.466 g

$$\text{Weight percentage of sulphur} = \frac{0.466 \times 32 \times 100}{0.2 \times 233} = 32$$

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 ($\text{Mg NH}_4 \text{PO}_4$), by addition of magnesia mixture ($\text{MgCl}_2 + \text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$)

- Magnesium ammonium phosphate is washed, dried and it is heated strongly to get magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$).



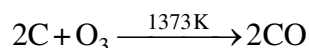
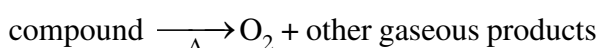
$$\% \text{P} = \frac{62}{222} \times \frac{\text{wt. of } \text{Mg}_2\text{P}_2\text{O}_7 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

- Phosphoric acid is precipitated as ammonium phospho molybdate $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3$ by adding ammonia and ammonium molybdate. (Molecular mass of $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 = 1877$)

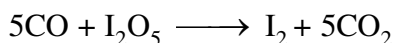
$$\% \text{P} = \frac{31}{1877} \times \frac{\text{wt of } (\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 \text{ formed}}{\text{wt of organic compound}} \times 100$$

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 Aluisse's method.
- A known amount of organic compound is subjected to pyrolysis in a stream of nitrogen.
- The mixture of gaseous products containing oxygen is passed over red-hot coke, then all the oxygen is converted in to carbon monoxide.



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



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 .

$$\% \text{ of oxygen} = \frac{16}{44} \times \frac{\text{wt. of } \text{CO}_2 \text{ formed}}{\text{wt. of organic compound}} \times 100$$

Ex.15 0.2g of an organic compound on analysis gave 0.147g of carbondioxide, 0.12g of water and 74.6 c.c of nitrogen at S.T.P. Calculate the weight percentages of constituents.

Sol. Weight of compound = W = 0.2g

Weight of CO_2 = W_1 = 0.147g

Weight of H_2O = W_2 = 0.12g

Volume of N_2 at STP = 74.6 c.c.

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

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

$$\% \text{ of Nitrogen} = \frac{W_2 \times 28 \times 100}{W \times 22400} = \frac{74.6}{8 \times 0.2} = 46.63\%$$

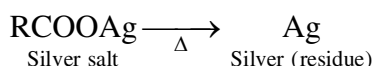
Remaining is oxygen.

$$\% \text{ of Oxygen} = 100 - (\%C + \%H + \%N) = 100 - 73.33 = 26.67\%$$

Note : Presently estimation of elements (C,H and N) in organic compound is estimated by using CHN elemental analyser by taking very small amount of substance (1-3 mg), results are displayed on screen with in short time.

Chemical Methods used to Find Molecular Mass Silver Salt Method for Acids :

Organic acid form insoluble silver salts, which on heating undergoes decomposition to leave a residue of metallic silver.



$$\frac{\text{Equivalent of silver salt}}{\text{Equivalent wt. of silver}} = \frac{\text{mass of silver salt}}{\text{Mass of silver}}$$

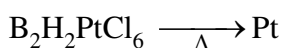
$$\frac{E + 108 - 1}{108} = \frac{\text{Mass of silver salt}}{\text{Mass of silver}}$$

$$E = \left[\frac{\text{Mass of silver salt}}{\text{Mass of silver}} \times 108 \right] - 107$$

Molecular weight of acid = Equivalent weight of acid (E) × basicity.

Platinic Chloride Method for Base

- Organic bases combines with chloroplatinic acid (H_2PtCl_6) to form insoluble platinichloride, which on ignition gives metallic platinum.
- If 'B' is mono acidic base then formula of salt will be $B_2H_2PtCl_6$.



$$\frac{\text{Molecular mass of platinum salt}}{\text{Atomic mass of platinum}} = \frac{\text{Mass of platinum salt}}{\text{Mass of platinum}}$$

If E is equivalent weight of base then

$$\frac{2E + 410}{195} = \frac{\text{Mass of platinum salt}}{\text{Mass of platinum}}$$

$$E = \frac{1}{2} \left[\frac{\text{Mass of platinum salt}}{\text{Mass of platinum}} \times 195 - 410 \right]$$

Molecular mass of base = Equivalent mass of base \times acidity

Empirical formula

- The simplest whole number ratio between the atoms of various elements present in one molecule of a substance is called empirical formula.

Calculation of Empirical Formula

The steps involved are

- Divide mass percentage of each element by its atomic mass, gives relative number of atoms.
- Simplest ratio is obtained when the figures obtained is divided by lowest number.
- If the simplest ratio obtained is not a whole number ratio, then multiply all the figures with suitable integer to get simplest whole number ratio.
- Write symbols of various elements side by side with above numbers at the lower right corner of each, which is empirical formula of compound.

Molecular formula

The actual number of atoms present in one molecule of a substance is called molecular formula.

Molecular formula = (Empirical formula)_n

where

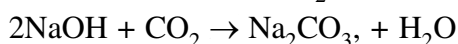
$$n = \frac{\text{Molecular wt of the compound}}{\text{Empirical formula weight of the compound}}$$

- If vapour density is given then molecular weight = $2 \times$ vapour density.

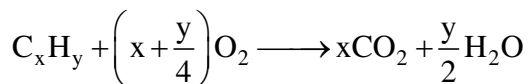
Determination of molecular formula of Gaseous Hydrocarbons (Eudiometry) :

- Eudiometry is a direct method used to find molecular formula of gaseous hydrocarbon without finding percentage composition of elements and its molecular weight.
- A known volume of gaseous hydrocarbon is mixed with excess of pure and dry oxygen in eudiometer tube placed inverted in a trough of mercury.
- The mixture is exploded by passing an electric spark between platinum electrodes. As a result carbon and hydrogen of hydrocarbon are oxidised to CO_2 and H_2O vapour respectively.
- The tube is allowed to cool to room temperature, then water vapour is condensed in to liquid water, whose volume occupied is almost negligible. Thus the gaseous mixture left in eudiometer tube is CO_2 and O_2 .
- Caustic potash solution is then introduced in to eudiometer tube which absorbs CO_2 completely.

The gas left is unused O_2 -



- Decrease in volume on introducing KOH solution gives volume of CO_2 used.
- Some times volume of O_2 left unused is found by introducing pyrogallol and noting the decrease in volume.
- Let molecular formula of gaseous hydrocarbon is C_xH_y . On combustion one volume of it forms x' volumes of CO_2 & ' $y/2$ ' volumes of water vapour.



$$1 \text{ vol} \left(x + \frac{y}{4}\right) \quad \quad x \text{ vol} \quad \quad \frac{y}{2} \text{ vol}$$

- For 1 volume of hydrocarbon

$$\text{Volume of } \text{O}_2 \text{ used} = \left(x + \frac{y}{4}\right) \text{ vol}$$

$$\text{Volume of } \text{CO}_2 \text{ produced} = x \text{ vol.}$$

$$\text{Contraction on explosion and cooling} = 1 + \frac{y}{4} \text{ (volume of liquid water is neglected)}$$

Determination of Molecular mass by Victor Meyer's Method

- A known mass of the volatile substance is vapourised in victor meyer's method.
- The vapours obtained displaces an equal volume of air in to graduated tube.
- The volume of air displaced is calculated at STP condition by using $\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2}$

$$\text{Mol. mass of volatile substance} = \frac{\text{Mass of substance taken}}{\text{Volume of air displaced in ml at STP}} \times 22400$$

EXERCISE # O-1

1. For the Separation of two immiscible liquids which method (or apparatus) is used?
 (1) Chromatography (2) Fractionating column
 (3) Fractional distillation (4) Separating funnel **PO0001**
2. A mixture of benzene and chloroform is separated by
 (1) Sublimation (2) Separating funnel (3) Crystallization (4) Distillation **PO0002**
3. Which of the following has molecular weight of 92 ?
 (1) Toluene (2) Benzene (3) Methylene (4) Propene **PO0003**
4. Aniline is usually purified by
 (1) Chromatographic technique (2) Steam distillation
 (3) By addition of oxalic acid (4) Fractional crystallization **PO0004**
5. The most suitable method for separation of 1:1 mixture of ortho- and para- nitrophenols is
 (1) Sublimation (2) Chromatography (3) Crystallization (4) Steam distillation
PO0005
6. Distillation under reduced pressure is employed for
 (1) Benzene (2) Petrol
 (3) Glycerol (4) Organic compounds used in medicine **PO0006**
7. Impure glycerine is purified by
 (1) Steam distillation (2) Simple distillation
 (3) Vacuum distillation (4) None of the above **PO0007**
8. Absolute alcohol is prepared by
 (1) Fractional distillation (2) Kolbe's method
 (3) Azotropic distillation (4) Vacuum distillation
PO0008
9. The latest technique used for purification for organic compounds in
 (1) Chromatography (2) Vacuum distillation
 (3) Fractional distillation (4) Crystallisation **PO0009**
10. In paper chromatography
 (1) Moving phase is liquid and stationary phase is solid
 (2) Moving phase is liquid and stationary phase is liquid
 (3) Moving phase is solid and stationary phase is solid
 (4) Moving phase is solid and stationary phase is liquid **PO0010**

11. A is a lighter phenol and B is an aromatic carboxylic acid. Separation of mixture A and B can be carried out easily by using a solution of
(1) Sodium hydroxide (2) Sodium sulphate (3) Calcium chloride (4) Sodium bicarbonate
PO0011
12. In sodium fusion test of organic compounds the nitrogen of the organic compound is converted into
(1) Sodamide (2) Sodium cyanide (3) Sodium nitrite (4) Sodium nitrate
PO0012
13. The Lassaigne's extract is boiled with dil. HNO_3 before testing for halogens because
(1) Silver halides are soluble in HNO_3 (2) Na_2S and NaCN are decomposed by HNO_3
(3) Ag_2S is soluble in HNO_3 (4) AgCN is soluble in HNO_3 **PO0013**
14. In a Lassaigne's test for nitrogen, the blue colour is due to the formation of
(1) Potassium ferricyanide (2) Sodium cyanide
(3) Sodium ferrocyanide (4) Ferri-ferro cyanide **PO0014**
15. Which of the following compounds does not show Lassaigne test for nitrogen?
(1) Urea (2) Hydrazine (3) Phenyl hydrozone (4) Azo benzene
PO0015
16. When piece of human hair are heated strongly with soda lime smell of ammonia can be detected. which one of the following conclusion can be drawn from this observation.
(1) Ammonia is present in human hair (2) Ammonium salt is present in hair
(3) Hair contains amino acids (4) None of the above **PO0016**
17. The Beilstein test for organic compounds is used to detect
(1) Nitrogen (2) Sulphur (3) Carbon (4) Halogen **PO0017**
18. Which of the following is the best scientific method to test presence of water in liquid?
(1) Smell (2) Taste
(3) Use of litmus paper (4) Use of anhydrous copper sulphate **PO0018**
19. Liebig's method is used for the estimation of
(1) Nitrogen (2) Sulphur
(3) Carbon and hydrogen (4) Halogens **PO0019**
20. 0.765 gm of an acid gives 0.535 gm of CO_2 and 0.138 gm of H_2O . Then the ratio of percentage of carbon and hydrogen is
(1) 19:2 (2) 18:11 (3) 20:17 (4) 1:7 **PO0020**

21. Nitrogen is an organic compound can be estimated by
 (1) Kjeldahl's method only (2) Duma's method only
 (3) Both the methods (4) none of these methods **PO0021**
22. Dumas method involves the determination of nitrogen content in the organic compound in form of
 (1) NH_3 (2) N_2 (3) NaCN (4) $(\text{NH}_4)_2\text{SO}_4$ **PO0022**
23. In Kjeldahl's method, nitrogen present is estimated as
 (1) N_2 (2) NO (3) NH_3 (4) NO_2 **PO0023**
24. The percentage of sulphur in an organic compound whose 0.32g produces 0.233g of BaSO_4 [At.wt.Ba=137, S=32] is
 (1) 1.0 (2) 10.0 (3) 23.5 (4) 32.1 **PO0024**
25. 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% **PO0025**
26. If a compound on analysis was found to contain C=18.5%, H= 1.55 %, Cl=55.04% and Oxygen =24.81%, Then the empirical formula is
 (1) CHClO (2) CH_2ClO (3) $\text{C}_2\text{H}_2\text{OCl}$ (4) ClCH_2O **PO0026**
27. An organic compound contains C=40 %, H= 13.33% and N=46.6 %. Its empirical formula would be
 (1) CHN (2) $\text{C}_2\text{H}_2\text{N}$ (3) CH_4N (4) $\text{C}_3\text{H}_7\text{N}$ **PO0027**
28. Two elements X (atomic weight =75) and Y(atomic weight=16) combine to give a compound having 75.8% X. The formula of the compound is
 (1) XY (2) X_2Y (3) X_2Y_2 (4) X_2Y_3 **PO0028**
29. On analysis a saturated hydrocarbon is found to contain 83.70% carbon and 16.30% hydrogen. The empirical formula will be (at wt. of C=12, at. wt. of H=1)
 (1) C_3H_6 (2) C_3H_8 (3) C_3H_4 (4) C_6H_{12} **PO0029**
30. Empirical formula of a hydrocarbon containing 80% carbon and 20% hydrogen is
 (1) CH (2) CH_2 (3) CH_3 (4) CH_4 **PO0030**
31. An organic compound with C= 40% and H= 6.7% will have the empirical formula
 (1) CH_4 (2) CH_2O (3) $\text{C}_3\text{H}_6\text{O}_3$ (4) $\text{C}_2\text{H}_4\text{O}_2$ **PO0031**
32. In a hydrocarbon, mass ratio of hydrogen and carbon 1:3, the empirical formula of hydrocarbon is
 (1) CH_4 (2) CH_2 (3) C_2H (4) CH_3 **PO0032**
33. 8.24 g of a volatile liquid on vaporization gives 45 ml of vapours at NTP. What will be the vapour density of the substance? (Density of $\text{H}_2=0.089 \text{ g L}^{-1}$)
 (1) 95.39 (2) 39.95 (3) 99.53 (4) 59.93 **PO0033**

34. 0.1914 g of an organic acid is dissolved in about 20 ml of water. 25 ml of 0.12 N. NaOH is required for the complete neutralization of the acid solution. The equivalent weight of the acid is
 (1) 65.0 (2) 64.0 (3) 63.8 (4) 62.5 **PO0034**
35. The equivalent weight of an acid is equal to
 (1) Molecular weight x acidity (2) Molecular weight x basicity
 (3) Molecular weight /basicity (4) Molecular weight /acidity **PO0035**
36. Simple distillation can be used to separate liquids which differ in their boiling points at least by
 (1) 5°C (2) 10°C (3) 35 - 50°C (4) 100°C **PO0036**
37. Empirical formula of a compound is CH_2O . If its vapour density is 90, then the molecular formula of the compound is
 (1) $\text{C}_5\text{H}_{10}\text{O}_5$ (2) $\text{C}_3\text{H}_6\text{O}_3$ (3) $\text{C}_6\text{H}_{12}\text{O}_6$ (4) $\text{C}_4\text{H}_8\text{O}_4$ **PO0037**
38. The empirical formula of an acid is CH_2O_2 , the probable molecular formula of the simplest acid may be
 (1) CH_2O (2) CH_2O_2 (3) $\text{C}_2\text{H}_4\text{O}_4$ (4) $\text{C}_3\text{H}_6\text{O}_6$ **PO0038**
39. A gaseous hydrocarbon has 85% carbon and vapour density of 28. The possible formula of the hydrocarbon will be
 (1) C_3H_6 (2) C_2H_4 (3) C_2H_2 (4) C_4H_8 **PO0039**
40. a hydrocarbon has C=85.72% and remaining H. The hydrocarbon is
 (1) C_2H_4 (2) C_2H_6 (3) C_2H_2 (4) CH_4 **PO0040**
41. An organic compound containing carbon, hydrogen and oxygen contains 52.2 % carbon 13.04% hydrogen. Vapour density of the compound is 23. Its molecular formula will be
 (1) $\text{C}_2\text{H}_6\text{O}$ (2) $\text{C}_3\text{H}_8\text{O}$ (3) $\text{C}_4\text{H}_8\text{O}$ (4) $\text{C}_5\text{H}_{10}\text{O}$ **PO0041**
42. percentage of Se(at.wt 78.4) in peroxidase anhydrase enzyme is 0.5% by weight, then minimum molecular weight of peroxidase anhydrase enzyme is
 (1) 1.568×10^4 (2) 1.568×10^3
 (3) 15.68 (4) 2.136×10^4 **PO0042**
43. The empirical formula of a compound is CH_2 . One of the compound has a mass of 42 g. its molecular formula is
 (1) CH_2 (2) C_2H_2 (3) C_3H_6 (4) C_3H_8 **PO0043**
44. In a compound C, H and N atoms are present in 9:1:3.5 by weight. If molecular weight of the compound is 108, then the molecular formula of the compound is
 (1) $\text{C}_2\text{H}_6\text{N}_2$ (2) $\text{C}_3\text{H}_4\text{N}$ (3) $\text{C}_6\text{H}_8\text{N}_2$ (4) $\text{C}_9\text{H}_{12}\text{N}_3$ **PO0044**

45. the following is the percentage composition of a compound. Na=16.08%, C=4.19% , O=16.78% , and $H_2O = 62.95\%$. Its molecular formula is
 (1) Na_2CO_3 (2) $Na_2CO_3 \cdot H_2O$ (3) $Na_2CO_3 \cdot 10H_2O$ (4) $Na_2CO_3 \cdot 5H_2O$ **PO0045**
46. An organic compound contains 49.3% carbon, 6.84% hydrogen and its vapour density is 73. Empirical Formula of the compound is
 (1) $C_3H_5O_2$ (2) $CH_{10}O_4$ (3) $C_3H_{10}O_2$ (4) $C_4H_{10}O_4$ **PO0046**
47. Molecular mass of a volatile substance may be obtained by
 (1) Kjeldahl's method (2) Duma's method
 (3) Victor meyer's method (4) Liebig's method **PO0047**
48. The most suitable method of separation of 1:1 mixture of ortho and para nitro phenol is
 (1) Distillation (2) Crystallization (3) Sublimation (4) Chromatography **PO0048**
49. Kjeldahl's method can not be used for the estimation of Nitrogen in
 (1) Pyridine (2) Nitro compounds (3) Azo compounds (4) All **PO0049**
50. In Kjeldahl's method nitrogen present is estimated as
 (1) N_2 (2) NH_3 (3) NO_2 (4) None **PO0050**

PURIFICATION AND CHARACTERISATION OF ORGANIC COMPOUNDS

1. Ans. 4	11. Ans. 4	21. Ans. 3	31. Ans. 2	41. Ans. 1
2. Ans. 4	12. Ans. 2	22. Ans. 2	32. Ans. 1	42. Ans. 1
3. Ans. 1	13. Ans. 2	23. Ans. 3	33. Ans. 4	43. Ans. 3
4. Ans. 2	14. Ans. 4	24. Ans. 2	34. Ans. 3	44. Ans. 3
5. Ans. 4	15. Ans. 2	25. Ans. 1	35. Ans. 3	45. Ans. 3
6. Ans. 3	16. Ans. 3	26. Ans. 1	36. Ans. 3	46. Ans. 1
7. Ans. 3	17. Ans. 4	27. Ans. 3	37. Ans. 3	47. Ans. 3
8. Ans. 3	18. Ans. 4	28. Ans. 4	38. Ans. 2	48. Ans. 1
9. Ans. 1	19. Ans. 3	29. Ans. 2	39. Ans. 4	49. Ans. 4
10. Ans. 2	20. Ans. 1	30. Ans. 3	40. Ans. 1	50. Ans. 2