

CHAPTER - 11

PURIFICATION AND CHARACTERISATION OF ORGANIC COMPOUNDS

Syllabus

Crystallization, Sublimation, distillation, differential extraction and chromatography- principles and their applications.

Qualitative analysis - Detection of nitrogen, sulphur, phosphorus and halogens

Quantitative Analysis(basic principles only) :- Estimation of carbon, hydrogen, nitrogen, halogens, sulphur, phosphorus.

Calculations of empirical and molecular formula: - Numerical problems in organic quantitative Analysis

METHODS OF PURIFICATION OF ORGANIC COMPOUNDS

When an organic compound is synthesised or extracted from a natural source, it is essential to purify it in order to study its properties. Methods of purification are based on the nature of the organic compound and the impurities present in it. The purity of a compound is ascertained by determining its melting or boiling point. Most of the pure compounds have sharp melting points and boiling points. Impurities generally lowers the melting point and elevates the boiling point of the substance.

Sublimation

Sublimation is defined as a process in which solid converts into gas directly without converting into liquid. The purification technique based on sublimation and is used to separate sublimable compounds from non-sublimable impurities

Common examples for sublimable organic solids are

- 1) Naphthalene
- 2) Anthracene
- 3) Benzoic acid
- 4) Phthalic anhydride
- 5) Camphor
- 6) Anthraquinone

Crystallisation

The process of formation of pure crystals of a compound by cooling a hot saturated solution of an impure sample of the compound in a suitable solvent is called crystallisation. This is one of the most commonly used techniques of purification of solid organic compounds. It is based on the difference of solubilities of the compound and the impurities in a suitable solvent.

Here a suitable solvent is added to the solid mixture, so that only one compound is soluble. The insoluble component is removed by filtration. The solution of the other component is the filtrate. The filtrate is concentrated by evaporation and solid will crystallise from the concentrated solution

For example, A mixture of urea and naphthalene can be separated by this technique. Here the suitable solvent is water. A mixture of benzoic acid and naphthalene can be separated by using hot water as the solvent.

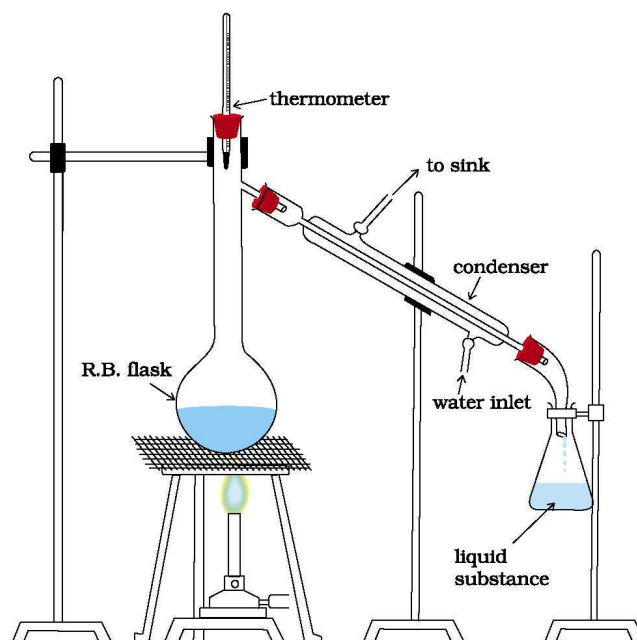
Distillation

Distillation is the process of heating a liquid to form vapour and then cooling the vapour to get back the liquid. The liquid obtained by condensing the vapour in the process of distillation is called distillate.

Simple Distillation

This method is used if there is sufficient difference in the boiling points of volatile liquids. (B.P. differ by atleast 30°C)

Liquids having different boiling points vapourise at different temperature. The vapours are cooled and the liquids so formed are collected separately

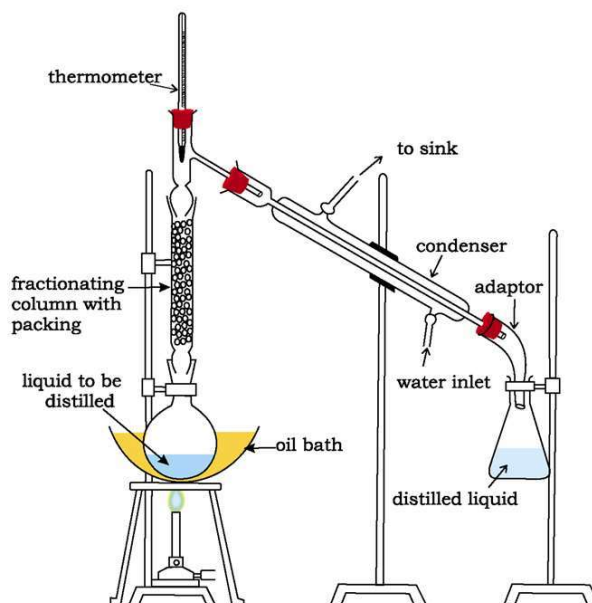


The liquid mixture is taken in a round bottom flask and heated carefully. On boiling the vapours of lower boiling component are formed first. The vapours are condensed by using a condenser and liquid is collected in a receiver. The vapours of high boiling component form later and the liquid can be collected separately.

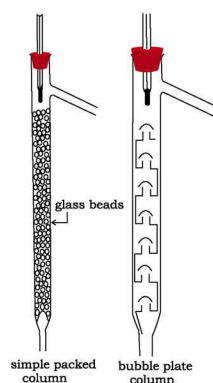
For example, A mixture of ether and toluene. B. P. of ether is 35°C , while that of toluene is 110°C . When the liquid mixture is heated to 35°C ether boils and the vapours are collected and condensed separately. Another example is a mixture chloroform(b.p. 334K) and aniline(b.p. 457K)

Fractional Distillation

The method is used to separate two volatile liquids when the difference in their boiling points is small ($10 - 15^{\circ}\text{C}$)



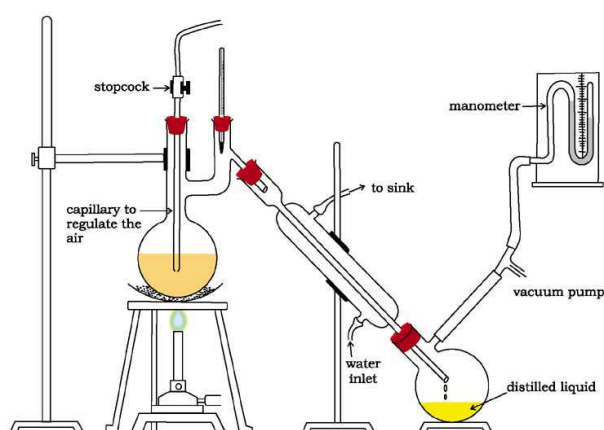
A fractionating column is fitted over the mouth of the distilling flask. A fractionating column provides many surfaces for heat exchange between ascending vapours and the descending liquid and as a result repeated evaporation and condensation occur throughout the fractionating column. The vapours thus become richer in low boiling component. The vapours of the low boiling component ascend to the top of the column. On reaching the top, the vapours become pure in low boiling component and pass through the condenser and the pure liquid is collected in a receiver. The remaining liquid in the distillation flask gets enriched in high boiling component. Each successive condensation and vapourisation unit in fractionating column is called a theoretical plate



Applications of fractional distillation is to separate different fractions of crude oil in petroleum industry.

Distillation under reduced pressure

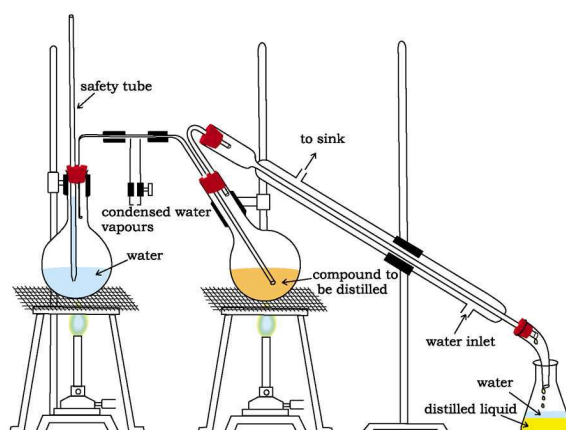
The Boiling point of a pure liquid is the temperature at which its vapour pressure becomes equal to external pressure. Depending upon the pressure applied, any given liquid have infinite number of boiling points. By lowering the external pressure it is possible to lower not only the boiling point but also its value to a level at which liquid vapourises without decomposition. Thus high boiling liquids as well as liquids which decompose at or below their normal boiling point are generally distilled at a temperature lower than their normal boiling point by reducing the external pressure on their surface. The pressure is reduced with the help of a water pump or vacuum pump.



Glycerol can be separated from spent lye in soap industry by using this technique. Spent lye is an alkaline solution containing soap and glycerol. Glycerol which decomposes at its boiling point(563K) can be distilled without decomposition at 453K under 12mm Hg pressure

Steam distillation

This technique is applied to separate substance which are steam volatile and are immiscible with water.



In steam distillation, steam from a steam generator is passed through heated flask containing the liquid to be distilled. The mixture of steam and the volatile organic compound is passes over the

condensor, condensed and collected in a receiver. The distillate contains desired substance and water which can be easily separated with help of a separating funnel.

In steam distillation, the liquid boils when the sum of vapour pressures due to the organic liquid (P_1) and that due to water (P_2) becomes equal to the atmospheric pressure is $P_1 + P_2 = P$. Since P_1 is lower than P , the organic liquid vapourises at lower temperature than its boiling point.

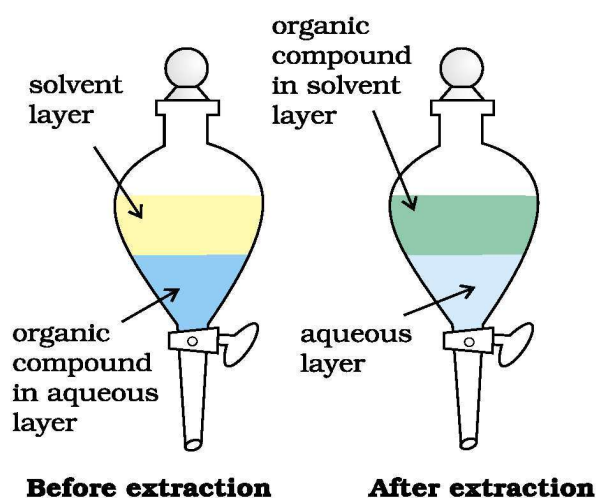
Aniline is separated by this technique from aniline- water mixture. Examples of organic substances that are purified by steam distillation

- 1) Aniline
- 2) Nitrobenzene
- 3) Bromobenzene
- 4) Salicylaldehyde
- 5) o- nitrophenol
- 6) o- hydroxyacetophenone
- 7) Essential oils like lemon grass oil

From a mixture of ortho and para nitrophenols the ortho isomer can be separated by steam distillation because only the ortho isomer is steam volatile while para is not.

Differential extraction

This method is based on the different solubilities of an organic compound in different solvents. If a compound is soluble in water but more soluble in an organic solvent, it is separated by shaking it with an organic solvent. As a result, the organic compound gets dissolved into an organic solvent which can be extracted from water.



Chromatography

Technique used to separate mixtures into their components, purify compounds and also to test the purity of compounds

Terms involved in chromatography

1) Stationary phase:- The phase in chromatography which is stationary with respect to the components of a mixture is called stationary phase. The mixture of substance is applied on to a stationary phase, which may be solid or a liquid

2) Mobile phase or Eluent: - The phase which moves along with the component of mixture is called a mobile phase or eluent. Mobile phase may be 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

Depending upon the mode of separation, chromatographic method can be broadly divided into

- a) Adsorption chromatography
- b) Partition chromatography

1) Adsorption chromatography

Adsorption:- The process of accumulation of molecules on the surface of liquid or solid substance rather than in bulk is called adsorption. The substance on which adsorption takes place is called adsorbent.

Adsorption chromatography is based on the fact that the different compounds are adsorbed on an adsorbent to different degree. Commonly used adsorbents are silica gel and alumina.

When a mobile phase (ie solvent) is allowed to move over a stationary phase (adsorbent), the components of the mixture move by varying distances over the stationary phase

Stationary phase:- Solid

Mobile phase: - Liquid

Column chromatography and thin layer chromatography are two examples for adsorption chromatography

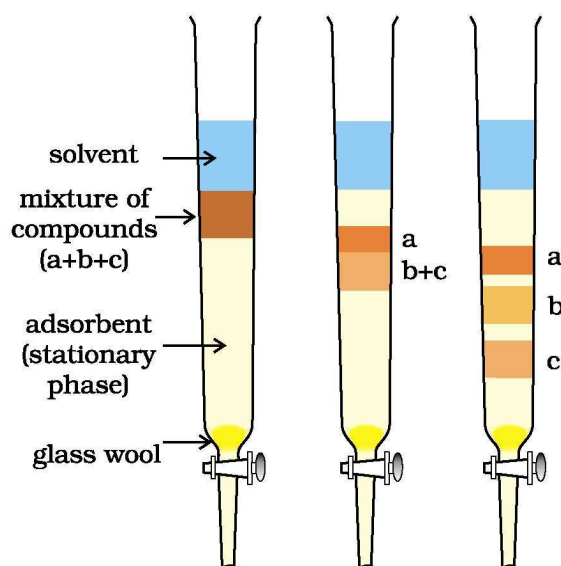
Column chromatography

Adsorbent (eg- silica gel) packed in a glass tube - Stationary phase.

The mixture adsorbed on adsorbent is placed on the top of the adsorbent column packed in a glass tube. An appropriate eluent is allowed to flow down the column slowly. Depending upon the degree to which the compounds are adsorbed complete separation takes place.

[Note:- The process of dissolving out the components from the adsorbent is called elution and the solvent used is termed as eluent]

Most readily adsorbed substances are retained near the top and other come down to various distances in the column.



Thin Layer Chromatography(TLC)

TLC involves separation of substances of a mixture over a thin layer of an adsorbent coated on glass plate(about 0.2mm thick)

A thin layer (about 0.2mm thick) of an adsorbent (silica gel or alumina) is spread over a glass plate(called thin layer chromatography plate or chromaplate)

The solution of the mixture is applied as a small spot about 2cm above one end of the TLC plate. The glass plate is then placed in a closed jar containing 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.

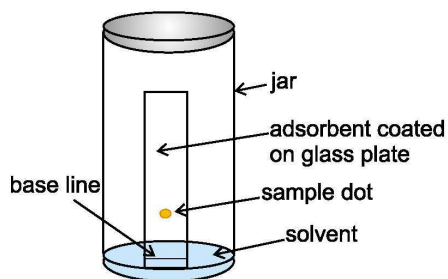
The relative adsorption of each component of the mixture is expressed in terms of its retardation factor (R_f value)

$$R_f = \frac{\text{Dis tan ce moved by the subs tan ce from the base line}}{\text{Dis tan ce moved by the solvent from the base line}}$$

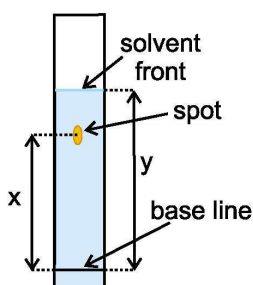
The spots of coloured compounds are visible on TLC plate due their original colour. The spots of colourless compounds, which are invisible to the eye but fluoresce in ultraviolet light, can be detected by putting the plate under UV light. Another detection technique is to place the plate in a covered jar containing few crystals of iodine. Spots of compounds which absorb iodine will show up as brown spots.

Sometimes an appropriate reagent may be sprayed on the plate.

For eg:- Aminoacids may detected by spraying the plate with ninhydrin solution.



*Thin layer chromatography.
Chromatogram being developed.*



Partition chromatography

Partition chromatography is based on continous differential partitioning of components of a mixture between stationary phase and mobile phases. Paper chromatography is a type of partition chromatography.

Paper chromatography

In paper chromatography, a special quality paper known as chromatography paper is used. Chromatography paper contains water trapped in it acts as the stationary phase

The solution of the mixture to be separated is applied as a small spot at the base of chromatography paper. It is suspended in a suitable solvent. This solvent acts as mobile phase. Solvent rises up the paper. After some time the spots of the separated coloured compounds are visible at different heights from the position of initial spot on the chromatogram (The paper strip so developed is known as chromatogram). The coloured components of a mixture are identified by their R_f value which can be given as

$$R_f = \frac{\text{Dis tan ce moved by the subs tan ce from the base line}}{\text{Dis tan ce moved by the solvent from the base line}}$$

In case of coloured compound, the spot is observed by the use of an appropriated spray reagent.

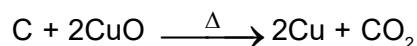
QUALITATIVE ANALYSIS OF ORGANIC COMPOUNDS

Identification of elements

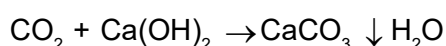
I) Detection of Carbon and Hydrogen

Experiment:- Organic compound + Copper(II) oxide $\xrightarrow{\text{Heat}}$

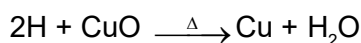
♦ Carbon (in organic compound) $\xrightarrow{\text{oxidation}}$ CO_2



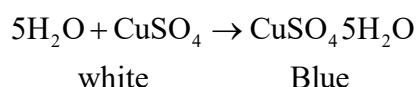
CO_2 tested with lime water($\text{Ca}(\text{OH})_2$) which develops turbidity



♦ Hydrogen(in organic compound) $\xrightarrow{\text{oxidation}}$ H_2O



H_2O tested with anhydrous copper sulphate which turns blue

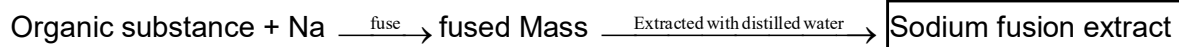


II) Detection of other Elements

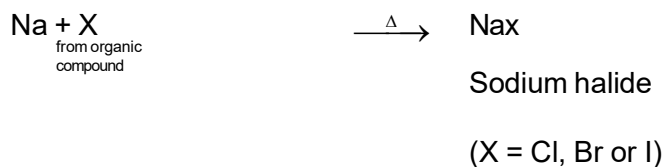
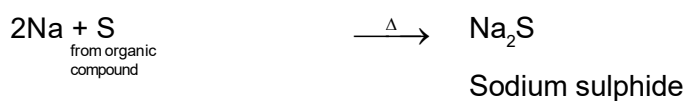
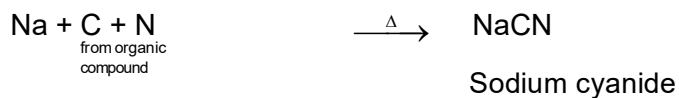
N, S, X(Cl, Br, I) present in an organic compound are detected by Lassaigne's test

Lassaignes test

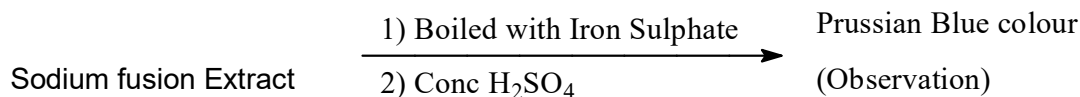
Preparation of Lassaignes Extract(Sodium fusion extract)



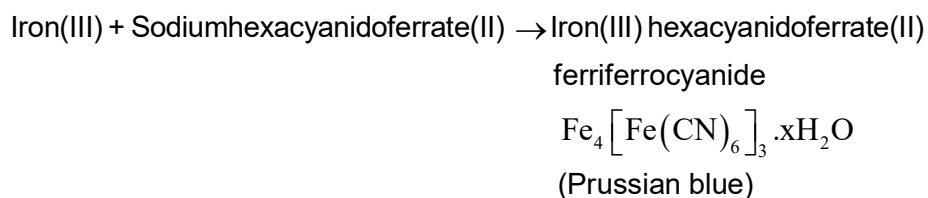
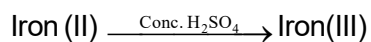
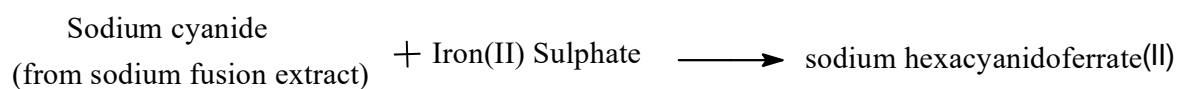
The elements present in the compound are converted from covalent form into ionic form by fusing the compound with sodium metal



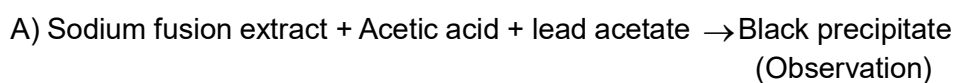
A) Test for Nitrogen



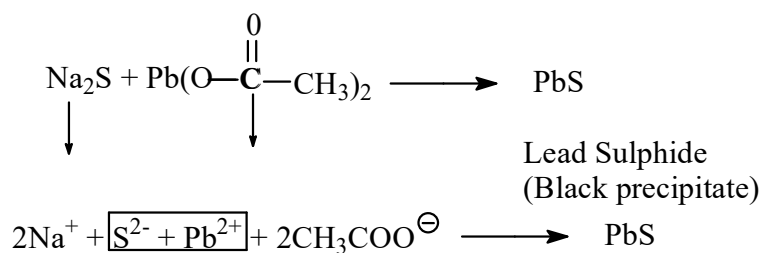
Inference: - Presence of Nitrogen



B) Test for Sulphur

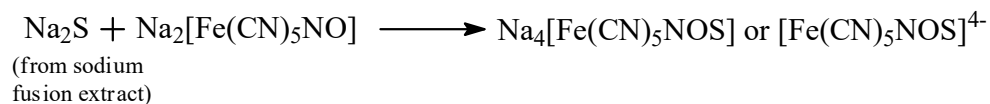


Inference:- Presence of sulphur

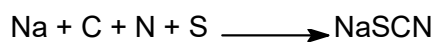


B) Sodium fusion extract + Sodium nitroprusside \longrightarrow Violet colour observation

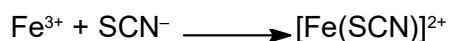
Inference:- presence of sulphur



In case, nitrogen and sulphur both are present in an organic compound, sodium thiocyanate is formed

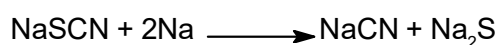


It gives blood red colour and no prussian blue since there are no free cyanide ions



Blood red

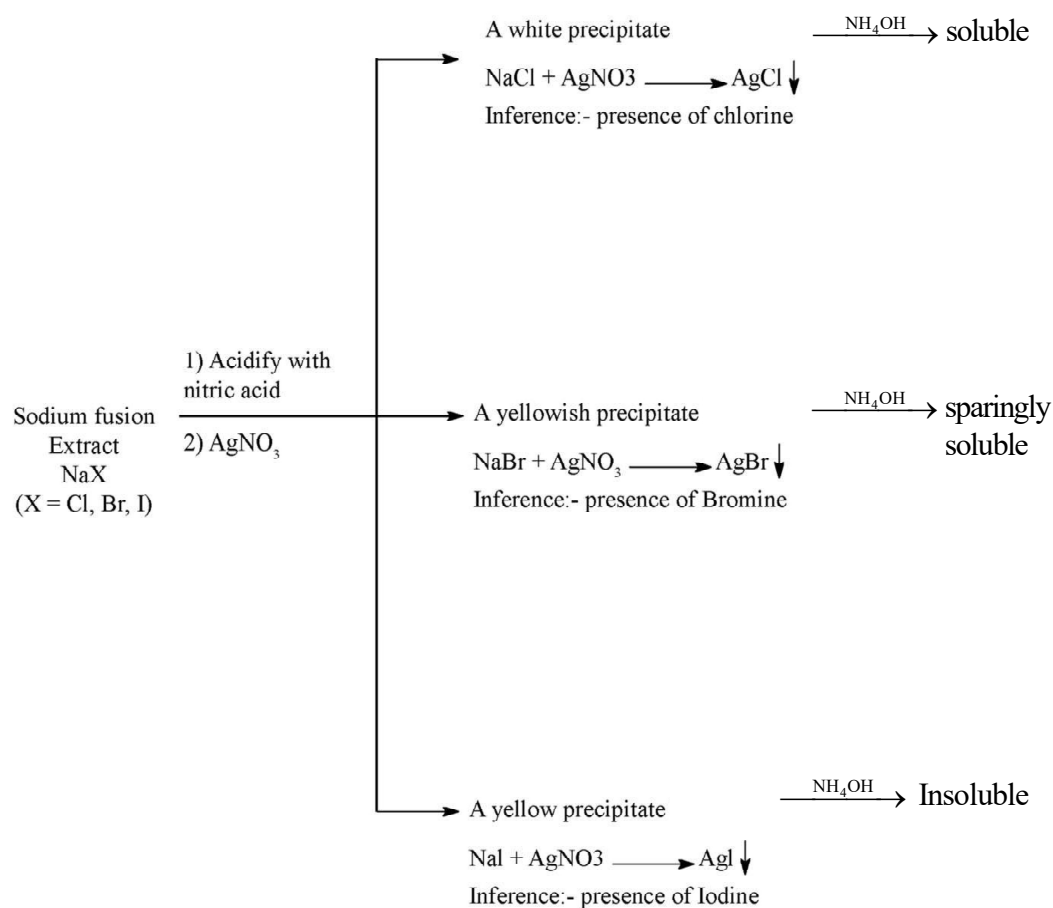
If sodium fusion is carried out with excess of sodium, the thiocyanate decomposes to yield cyanide and sulphide. These ions give their usual tests



Note :- Some compounds that may produce blood red colouration

- | | |
|---------------|---------------------|
| 1) Thiourea | 2) Sulphanilic acid |
| 3) Saccharin | 4) Cystein |
| 5) Methlonine | |

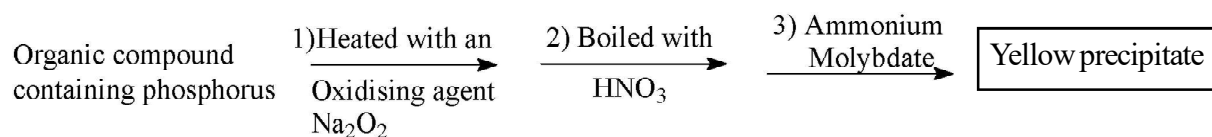
C) Test for Halogens



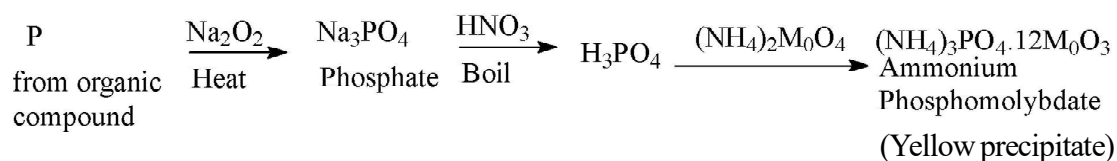
If nitrogen or sulphur is also present in the compound, the sodium fusion extract is first boiled with conc HNO₃ to decompose cyanide or sulphide of sodium formed during lassaigne's test. These ions would otherwise interfere with silver nitrate test for halogens.

We cant detect fluorine by this test

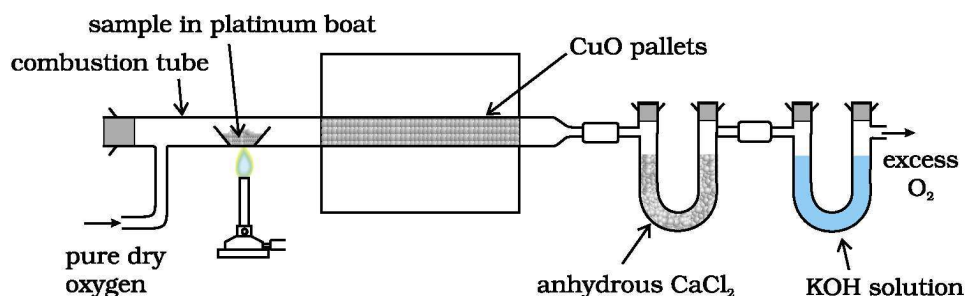
Reason:- AgF is water soluble so will not give any precipitate on adding AgNO₃ solution.

Test for phosphorus

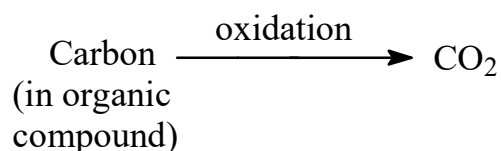
Inference:- presence of phosphorus

**QUANTITATIVE ANALYSIS**

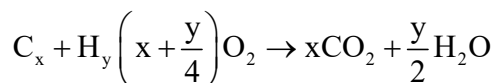
- ◆ Determination of mass percent of elements present in a compound
- ◆ Mass percent of elements is required for the determination of empirical and molecular formula

1) Estimation of Carbon and Hydrogen(Liebig's combustion method)

A known mass(mg) of an organic compound is burnt in the presence of excess of oxygen and copper(II) oxide



Hydrogen $\xrightarrow{\text{oxidation}}$ H_2O
(in organic compound)



Water and carbon dioxide formed on oxidation of substance are absorbed in anhydrous CaCl_2 and potassium hydroxide solutions respectively contained in U- tubes. The increase in mass of CaCl_2 tube gives the mass of water. Increase in mass of KOH tube gives the mass of CO_2 . From this we can calculate the percentages of carbon and hydrogen.

$$\text{Percentage of carbon in the organic compound} = \frac{12 \times \text{Mass of } \text{CO}_2 \text{ produced} \times 100}{44 \times \text{Mass of organic compound taken}}$$

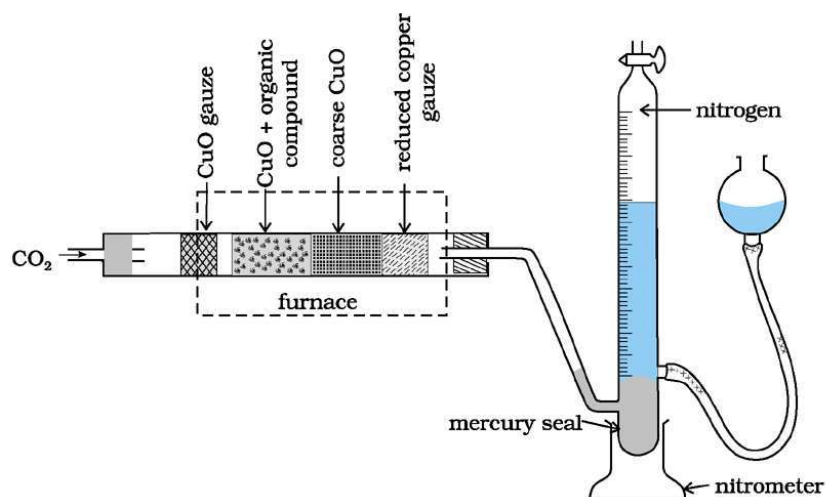
$$\text{Percentage of hydrogen in the organic compound} = \frac{2 \times \text{Mass of water produced} \times 100}{18 \times \text{Mass of organic compound taken}}$$

2) Estimation of Nitrogen

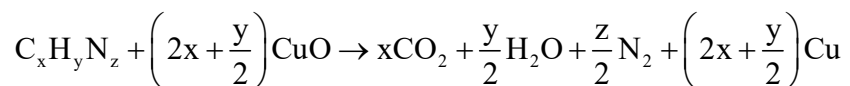
Two methods for estimation of nitrogen

- 1) Duma's Method
- 2) Kjeldahl's method

1) Duma's Method



Nitrogen containing organic compound, when heated with copper oxide in an atmosphere of CO_2 yields free nitrogen, CO_2 and water



Traces of Nitrogen oxides $\xrightarrow[\text{(Reduction)}]{\text{Heated copper gauze}}$ Nitrogen
(If any)

The mixture of gases coming out of the combustion tube contains CO_2 , water vapour and N_2 . This gaseous mixture is collected over KOH solution in a Schiff's nitrometer. KOH solution will absorb CO_2 and water vapour. Nitrogen gas gets collected at the upper part of the nitrometer (upper portion of a graduated tube). Here the volume of N_2 gas collected can be measured.

Calculation

Mass of organic compound taken = m grams

Volume of nitrogen gas collected = V_1 mL

Room temperature at which nitrogen gas is collected = T_1 K

In this situation the nitrogen gas is moist due to the presence of small quantities of water vapour caused by evaporation. Total pressure exerted by the moist gas is the combined pressure exerted by dry N_2 gas and water vapour.

Pressure exerted by dry N_2 gas = Atmospheric pressure at which N_2 is collected

(P_1) – Aqueous tension at Room temperature
(Pressure of water vapour at Room temperature)

Pressure exerted by water vapour is called aqueous tension

1 mol of every gas at STP (273K and 1atm pressure) occupies a volume of 22.4L or 22400 mL.

Applying the combined gas equation $\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2}$

$$\frac{\text{Pressure exerted by dry } \text{N}_2 (P_1) \times \text{Volume of } \text{N}_2 \text{ gas collected } (V_1)}{\text{Room Temperature at which } \text{N}_2 \text{ gas is collected } (T_1)} = \frac{760 \times \text{Volume of } \text{N}_2 \text{ gas at STP}}{273}$$

$$\text{Volume of } \text{N}_2 \text{ gas at STP} = V_2 = \frac{P_1 V_1}{T_1} \times \frac{273}{760}$$

Molar mass of N_2 gas is 28g

22400 ml N_2 gas at STP weighs 28g Nitrogen

1ml N_2 gas at STP weigh $\frac{28}{22400}$ g nitrogen

V_2 mL N_2 gas at STP weigh $\frac{28 \times V_2}{22400}$ g nitrogen

ie, Mass of V_2 mL of N_2 gas at STP = $\frac{28 \times V_2}{22400}$ g

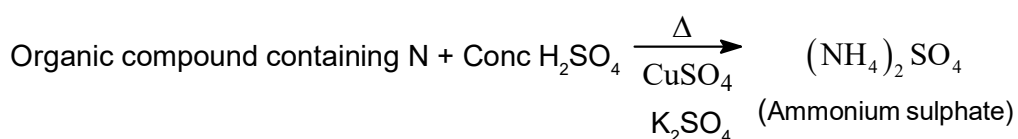
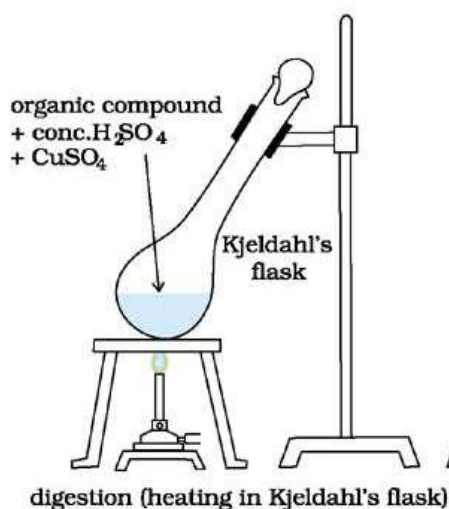
$$\% \text{ of Nitrogen in the organic compound} = \frac{\text{Mass of nitrogen}}{\text{Mass of organic compound}} \times 100 = \frac{28 \times V_2 \times 100}{22400 \times m}$$

$$\% \text{ of Nitrogen in the organic compound} = \frac{28 \times \text{Volume of } N_2 \text{ gas at STP} \times 100}{22400 \times \text{Mass of organic compound taken}}$$

2) Kjeldahl's Method

Nitrogen containing organic compound is treated with concentrated H_2SO_4 to get ammonium sulphate which liberates ammonia on treating with NaOH; ammonia is absorbed in known volume of standard acid.

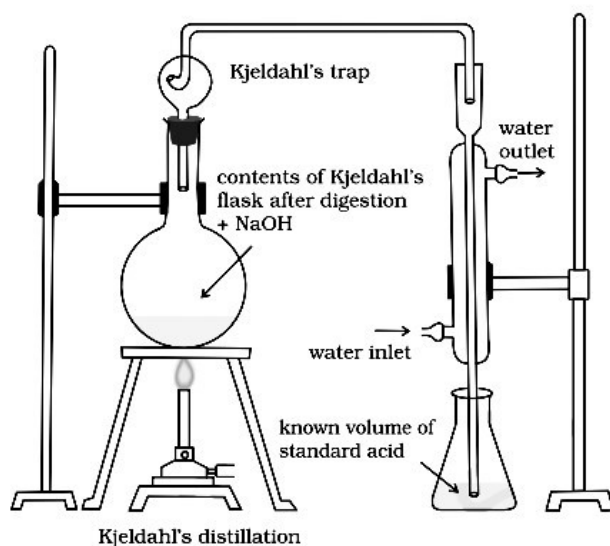
1) Digestion(Heating in Kjeldahl's flask)



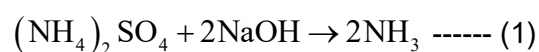
$\text{K}_2\text{SO}_4 \longrightarrow$ To raise the boiling point of H_2SO_4

$\text{CuSO}_4/\text{Hg} \longrightarrow$ Acts as catalyst

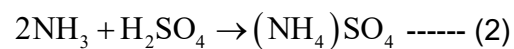
2) Kjeldahl's distillation



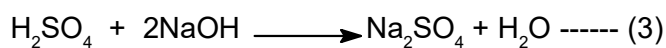
Contents of Kjeldahl flask after digestion + NaOH $\xrightarrow{\Delta}$ Evolution of NH_3



The liberated ammonia gas is absorbed in an excess of standard solution of sulphuric acid



The amount of NH_3 produced (equation 1) is determined by estimating the amount of sulphuric acid consumed (equation 2). It is done by estimating unreacted H_2SO_4 left after the absorption of ammonia by titrating it with standard alkali solution (NaOH)



(Unreacted)

The difference between the initial amount of acid taken and that left after the above reaction (equation 3) gives the amount of acid reacted with ammonia

$$\text{Normality of H}_2\text{SO}_4 = N_1$$

$$\text{Volume of excess(total) H}_2\text{SO}_4 = V_1 \text{ mL}$$

$$\text{Normality of NaOH used for the titration of unreacted H}_2\text{SO}_4 = N_2$$

$$\text{Volume of NaOH used for the titration of unreacted H}_2\text{SO}_4 = V_2 \text{ mL}$$

$$\therefore \% \text{ of Nitrogen in the organic compound } = \frac{(N_1 V_1 - N_2 V_2) \times 14 \times 100}{1000 m}$$

m is the mass of the organic compound taken

Note: Normality = Molarity \times Basicity(for acids)

Normality = Molarity \times Acidity(for bases)

$$N_1 V_1 = \text{Miliequivalents of excess(total) H}_2\text{SO}_4 \text{ taken}$$

$$N_2 V_2 = \text{Miliequivalents of unreacted H}_2\text{SO}_4$$

$$[\text{Miliequivalents of unreacted H}_2\text{SO}_4 = \text{Miliequivalents of NaOH} = N_2 V_2]$$

$$\therefore N_1 V_1 - N_2 V_2 = \text{Miliequivalents of H}_2\text{SO}_4 \text{ neutralised by NH}_3$$

$$= \text{Normality of H}_2\text{SO}_4 \times \text{Volume of H}_2\text{SO}_4 \text{ used by NH}_3$$

$$\therefore \% \text{ Nitrogen in the organic compound}$$

$$= \frac{\text{Normality of H}_2\text{SO}_4 \times \text{Volume of H}_2\text{SO}_4 \text{ used by NH}_3 \times 14 \times 100}{1000 \times \text{Mass of organic compound taken}}$$

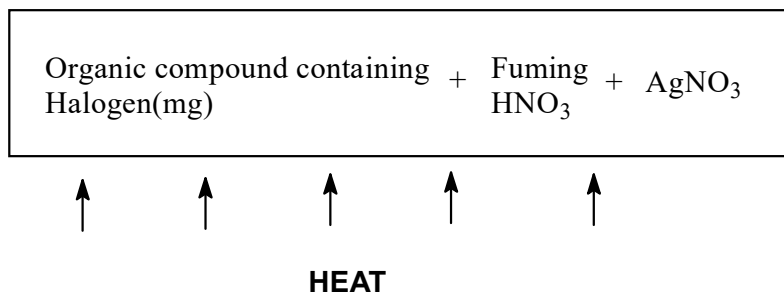
% N in the organic compound

$$= \frac{\text{Molarity of H}_2\text{SO}_4 \times \text{Basicity of H}_2\text{SO}_4 \times \text{Volume of H}_2\text{SO}_4 \text{ used by NH}_3 \times 14 \times 100}{1000 \times \text{Mass of organic compound taken}}$$

Estimation of Halogens

Carius Method

Mass of halogen containing organic compound = m gram

CARIUS TUBE

Carbon $\xrightarrow{\text{oxidation}}$ Carbon dioxide

Hydrogen $\xrightarrow{\text{oxidation}}$ Water

Halogen \longrightarrow Silver halide

X AgX↓

The precipitate (AgX) is Filtered \longrightarrow Washed \longrightarrow Dried \longrightarrow Weighed(m_1 g) AgX

Mass of AgX formed = m_1 g

1 mol of Agx contains 1mol of X

Mass of Halogen in m_1 g of AgX = $\frac{\text{Atomic mass of X} \times \text{Mass of Ag X formed}}{\text{Molecular mass of AgX.}}$

% of Halogen in the organic compound = $\frac{\text{Atomic mass of X} \times \text{Mass of AgX formed} \times 100}{\text{Molecular mass of AgX} \times \text{Mass of organic compound taken}}$

% of Chlorine = $\frac{35.5 \times \text{Mass of AgCl formed} \times 100}{143.5 \times \text{Mass of organic compound taken}}$

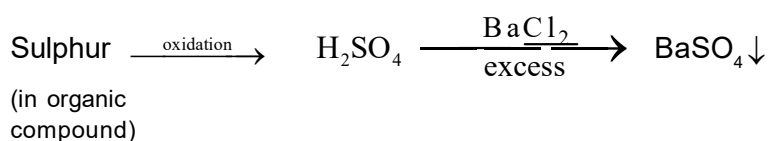
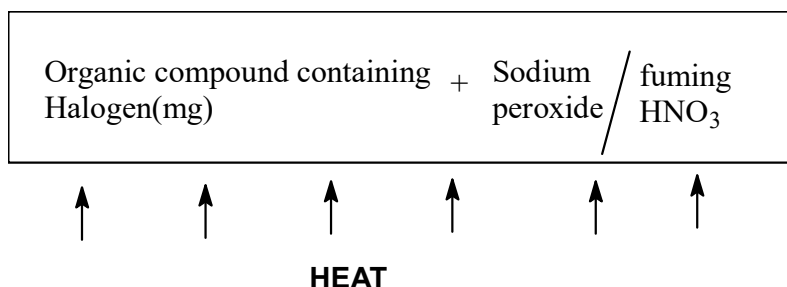
% of Bromine = $\frac{80 \times \text{Mass of AgBr formed} \times 100}{188 \times \text{Mass of organic compound taken}}$

% of Iodine = $\frac{127 \times \text{Mass of AgI formed} \times 100}{235 \times \text{Mass of organic compound taken}}$

Estimation of Sulphur**Carius Method**

Mass of Sulphur contain organic compound = mg

CARIUS TUBE

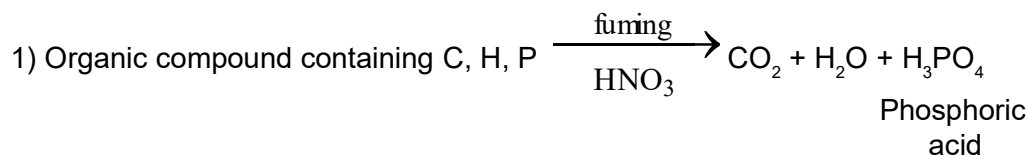


The precipitate (BaSO₄) is Filtered → Washed → Dried → Weighed (BaSO₄) (m₁g)

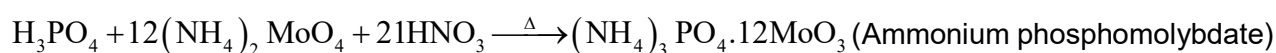
Mass of Barium sulphate formed = m₁g

$$\% \text{ of Sulphur in organic compound} = \frac{32 \times \text{Mass of BaSO}_4 \text{ formed} \times 100}{233 \times \text{Mass of organic compound taken}}$$

Estimation of phosphorus



Phosphoric acid is quantitatively precipitated as ammonium phosphomolybdate

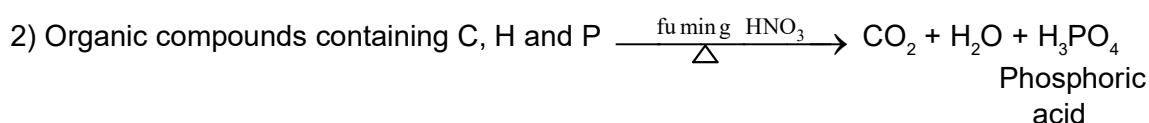


The precipitate → Filtered → Washed → Dried (m₁g)

The percentage of P can be calculated as follows

1 mol Ammonium phosphomolybdate contains 1 mol P.

$$\% \text{ of Phosphorus in the organic compound} = \frac{31 \times \text{Mass of } (\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3 \text{ formed} \times 100}{1877 \times \text{Mass of organic compound taken}}$$



Alternatively, phosphoric acid is quantitatively precipitated as Magnesium ammonium phosphate

(MgNH_4PO_4) by heating the solution with Magnesia mixture($\text{MgCl}_2 + \text{NH}_4\text{Cl} + \text{NH}_3$)

The white precipitate of magnesium ammonium phosphate is filtered, washed and dried. On Ignition it gets converted into Magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$), which is weighed to calculate the percentage of P as follows

1 mol $\text{Mg}_2\text{P}_2\text{O}_7$ contains 2 mol P.

$\text{Percentage of phosphorus in the organic compound} = \frac{62 \times \text{Mass of } \text{Mg}_2\text{P}_2\text{O}_7 \text{ formed} \times 100}{222 \times \text{Mass of organic compound taken}}$
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Estimation of oxygen

First Method

% of oxygen in the organic compound = 100 – (sum of the percentage of all other elements)

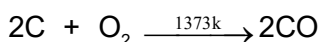
Percentage of Oxygen = 100 – (Percentage of C + percentage of H + -----)

Second Method

Aluise's Method

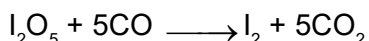
A definite mass of an organic compound(mg) is decomposed by heating in a stream of nitrogen gas. The mixture of gaseous products containing oxygen is passed over red hot coke when all the oxygen is converted to carbon monoxide. This mixture is passed through warm iodine pentoxide when carbon monoxide is oxidised to carbon dioxide producing iodine. The CO_2 produced is then absorbed in a previously weighed tube containing KOH solution. After the experiment, the mass increase of KOH tube will give the mass of CO_2 produced

Organic compound containing oxygen $\xrightarrow{\text{heat}}$ O_2 + other gaseous products



Red

hot coke



Final equation is $2\text{I}_2\text{O}_5 + 10\text{C} + 5\text{O}_2 \rightarrow 2\text{I}_2 + 10\text{CO}_2$

from $5\text{mol O}_2 \rightarrow 10\text{mol CO}_2$

ie $1\text{mol O}_2 \rightarrow 2\text{mol CO}_2$

88g of carbon dioxide is obtained if 32g oxygen is liberated.

m_1 g of carbon dioxide is obtained from $\frac{32 \times m_1}{88}$ g O_2

$\therefore \text{Percentage of oxygen in the organic compound} = \frac{32 \times \text{Mass of } \text{CO}_2 \text{ produced} \times 100}{88 \times \text{Mass of organic compound taken}}$
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CHN Elemental analyser

The elements, carbon, hydrogen and nitrogen present in a compound are determined by an apparatus known as CHN elemental analyser. The analyser requires only a very small amount of substance(1 - 3 mg) and displays the values on a screen within a short time.