

1. Details of Module and its structure

Module Detail	
Subject Name	Chemistry
Course Name	Chemistry 01 (Class XI, Semester - 2)
Module Name/Title	Organic Chemistry – Some Basic Principles and Techniques: Part 4
Module Id	kech_21204
Pre-requisites	Knowledge about sublimation, boiling point, melting point and solubility of organic compounds
Objectives	After going through this lesson, the learners will be able to: <ul style="list-style-type: none">• Learn about different methods of purification for organic compounds• Understand that solid compounds can be purified by sublimation or crystallization• Differentiate between normal distillation and fractional distillation for the purification of liquid compounds.• Explain qualitative analysis of carbon, hydrogen and heteroatoms present in organic compounds• Learn about quantitative analysis of organic compounds
keywords	Sublimation, Crystallization, Distillation, Chromatography Qualitative Analysis, Lassaigne's Method Quantitative Analysis

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1. Introduction

In this module you will learn about the various techniques of purification of organic compounds. You will also learn qualitative and quantitative analysis of organic compounds for determining their structures.

2. Methods of Purification of Organic Compounds

Any organic compound that is either extracted from a natural source or synthesized in the laboratory is not pure. It is essential to purify organic compound for the identification. There are

several methods used for the purification of organic compounds that are based on the nature of the compound and the impurity present in it. Finally, the purity of a compound is confirmed by determining its melting or boiling point. Most of the pure compounds have sharp melting points and boiling points. New methods of checking the purity of an organic compound are based on different types of chromatographic and spectroscopic techniques.

The common techniques used for purification are as follows:

- i. Sublimation
- ii. Crystallisation
- iii. Distillation
- iv. Differential extraction
- v. Chromatography

2.1 Sublimation

As you are aware, some solid substances on heating change from solid to vapour state without passing through liquid state. The purification technique based on the above principle is known as sublimation and is used to separate sublimable compounds from non-sublimable impurities. For example, iodine and naphthalene can be purified using sublimation.

2.2 Crystallisation

One of the most common techniques for the purification of solid organic compounds is crystallization. It is based on the difference in the solubilities of the compound and the impurities in a suitable solvent. The solvent for crystallisation is chosen such that the impure compound is sparingly soluble in it at room temperature but appreciably soluble at higher temperature. The solution is concentrated to get a nearly saturated solution. On cooling the solution, pure compound crystallises out and is collected by filtration. The filtrate (mother liquor) contains impurities as well as small quantity of the compound. Crystallisation can also be carried out in a mixture of solvents, if the compound is highly soluble in one solvent and very little soluble in another solvent. Some Impurities impart colour to the solution and are removed by adsorbing over activated charcoal. Repeated crystallization becomes necessary for the purification of compounds containing impurities of comparable solubilities.

2.3 Distillation

Distillation is very important method used to separate

- i. Volatile liquids from nonvolatile impurities and
- ii. The liquids having sufficient difference in their boiling points.

2.3.1 Simple distillation

Liquids having different boiling points form vapors at different temperatures. The vapours are cooled and the liquids so formed are collected separately. For example, chloroform (b.p 334 K) and aniline (b.p. 457 K) are easily separated by the technique of distillation (**Figure 1**). The liquid mixture is taken in a round bottom flask and heated carefully. On boiling, the component with lower boiling point (chloroform) will vaporize first. The vapours are then condensed by using a condenser and the liquid is collected in a receiver. At boiling point of any liquid the temperature of liquid mixture is not raised and once the whole of low boiling component is vaporized, then there is rise in temperature. Now, the vapours of higher boiling component (aniline) start forming and the liquid can be collected separately.

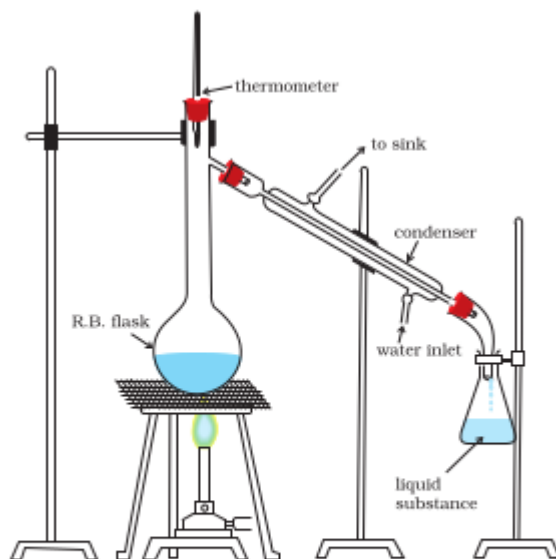


Figure 1. Simple distillation.

2.3.2 Fractional Distillation

If the difference in boiling points of two liquids is not much, simple distillation cannot be used to separate them. The vapours of such liquids are formed within the same temperature range and are condensed simultaneously. The technique of fractional distillation is used in such cases. In this technique, fractionating column is fitted over the mouth of the round bottom flask (Figure 2) and

vapours of a liquid mixture are passed through a fractionating column before condensation. The vapours rising up in the fractionating column become richer in more volatile component. By the time the vapours reach to the top of the fractionating column, these are rich in the more volatile component. Fractionating columns are available in various sizes and designs as shown in figure 3.

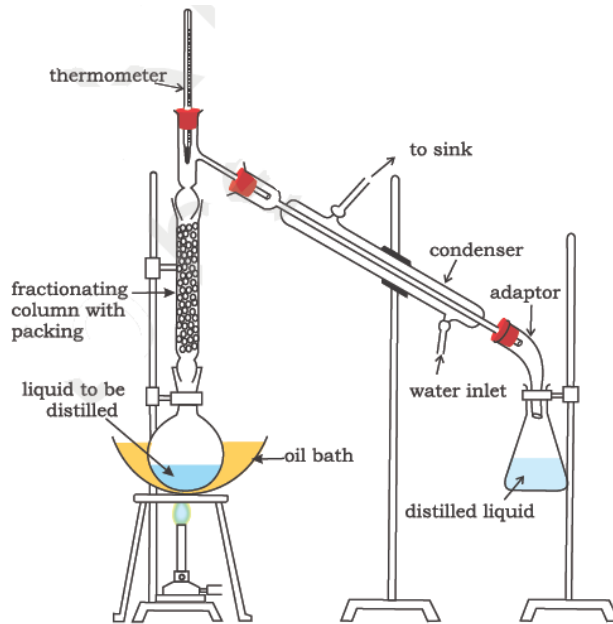


Figure 2. Fractional distillation

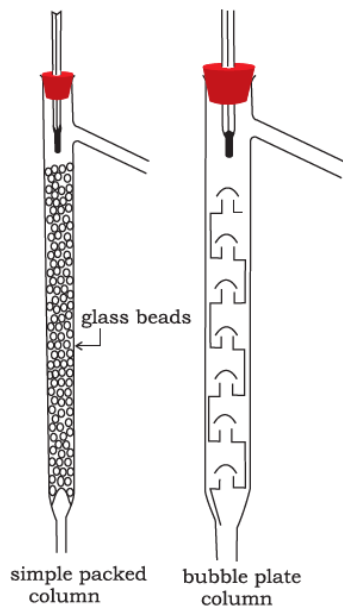


Figure 3. Different types of fractionating columns

A fractionating column provides many surfaces for heat exchange between the ascending vapours and the descending condensed liquid. Some of the condensing liquid in the fractionating

column obtains heat from the ascending vapours and revaporises. The vapours thus become richer in low boiling component. The vapours of 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. After a series of successive distillations, the remaining liquid in the distillation flask gets enriched in high boiling component. Each successive condensation and vaporisation unit in the fractionating column is called a theoretical plate.

Commercially, columns with hundreds of plates are available. One of the technological applications of fractional distillation is to separate different fractions of crude oil in petroleum industry. The vapours of lower boiling fraction reach the top of the column first followed by vapours of higher boiling fractions.

2.3.3 Distillation under reduced pressure

Liquids having very high boiling point and those, which decompose at or below their boiling point can be purified by distillation under reduced pressure. Such liquids are made to boil at a temperature lower than their normal boiling points by reducing the pressure on their surface. A liquid boils at a temperature at which its vapour pressure is equal to the external pressure. The pressure is reduced with the help of a water pump or vacuum pump (Figure 4). Glycerol can be separated from spent-lye in soap industry by using this technique.

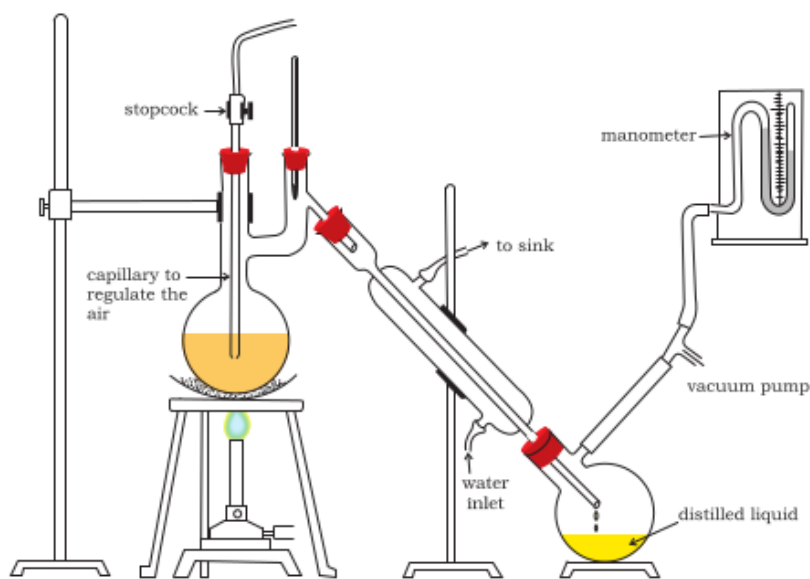


Figure 4. Distillation under reduced pressure

2.3.4 Steam Distillation

The liquids which are steam volatile and immiscible with water can be purified using steam distillation. In this technique, steam from a steam generator is passed through a heated flask containing the liquid to be distilled. The mixture of steam and the volatile organic compound is condensed and collected. The compound is later separated from water using 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 (P), i.e. $P = P_1 + P_2$. Since P_1 is lower than P , the organic liquid vaporises at lower temperature than its boiling point. Thus, if one of the substances in the mixture is water and the other, a water insoluble substance, then the mixture will boil close to but below, 373K. A mixture of water and the substance is obtained which can be separated by using a separating funnel.

Aniline is separated by this technique from aniline – water mixture (Figure 5).

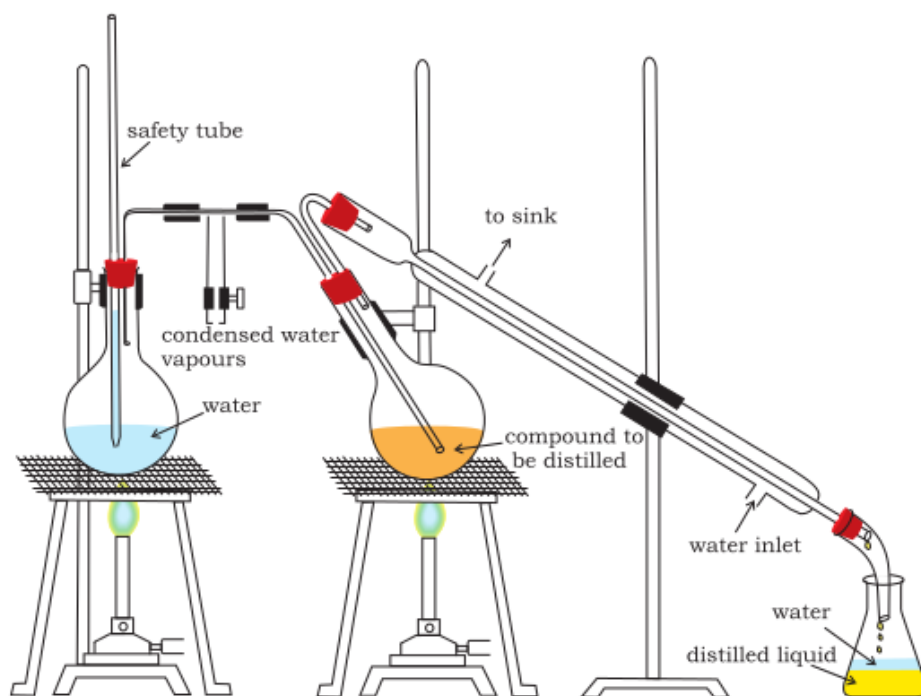


Figure 5. Steam distillation

2.4 Differential Extraction

Any organic compound present in an aqueous medium, it is separated by shaking it with an organic solvent in which it is more soluble than in water. The organic solvent and the aqueous solution should be immiscible with each other so that they form two distinct layers which can be separated by separating funnel. The organic solvent is later removed by distillation or by evaporation to get back the compound. Differential extraction is carried out in a separating

funnel as shown in figure 6. If the organic compound is less soluble in the organic solvent, a very large quantity of solvent would be required to extract even a very small quantity of the compound. The technique of continuous extraction is employed in such cases. In this technique same solvent is repeatedly used for extraction of the compound.

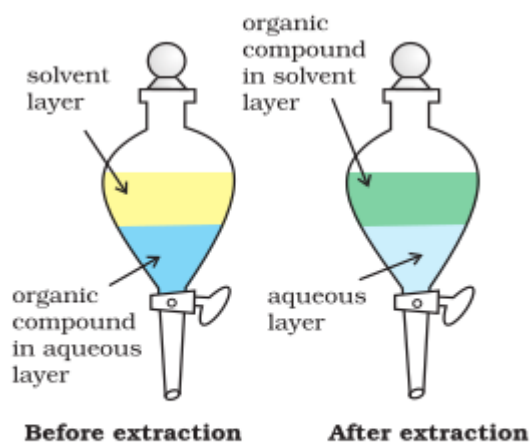


Figure 6. Differential extraction

2.5 Chromatography

Chromatography is an important technique extensively used to separate mixtures into their components, purify compounds and also test the purity of compounds. The name chromatography is based on the Greek word chroma, for colour since the method was first used for the separation of coloured substances found in plants. In this technique, the mixture of substances is applied onto a stationary phase, which may be a solid or a 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. The moving phase is called the mobile phase. Based on the principle involved, chromatography is classified into different categories. Two of these are: (1) Adsorption chromatography, and (2) Partition chromatography.

2.5.1 Adsorption Chromatography

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

1. Column chromatography
2. Thin layer chromatography

(1) Column Chromatography

Column chromatography involves separation of a mixture over a column of adsorbent (stationary phase) packed in a glass tube. The column is fitted with a stopcock at its lower end (Figure 7). The mixture adsorbed on adsorbent is placed on the top of the adsorbent column packed in a glass tube and then appropriate eluent which is a liquid or a mixture of liquids is allowed to flow down the column slowly. Depending upon the degree to which the compounds are adsorbed, complete separation takes place. The most readily adsorbed substances are retained near the top and others come down to various distances in the column (Figure 7).

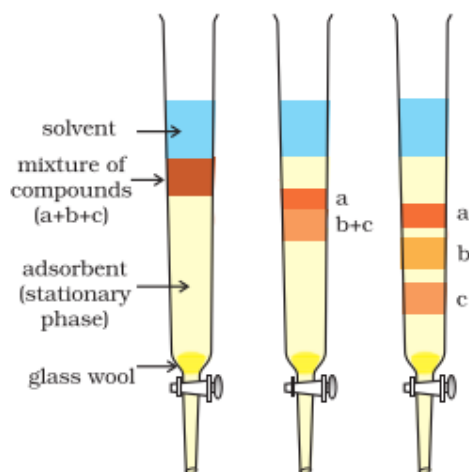


Figure 7. Column chromatography

(2) Thin Layer Chromatography

Thin layer chromatography (TLC) is another type of adsorption chromatography, which involves separation of substances of a mixture over a thin layer of an adsorbent coated on glass plate. A thin layer (about 0.2mm thick) of an adsorbent (silica gel or alumina) is spread over a glass plate of suitable size. The plate is known as thin layer chromatography plate (TLC plate) or chromaplate. The solution of the mixture to be separated is applied as a small spot about 2 cm above one end of the TLC plate. The glass plate is then placed in a closed jar containing the eluent (Figure 8a). As the eluent rises up the plate, the components of the mixture move up along with the eluent 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 (Figure 8b).

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

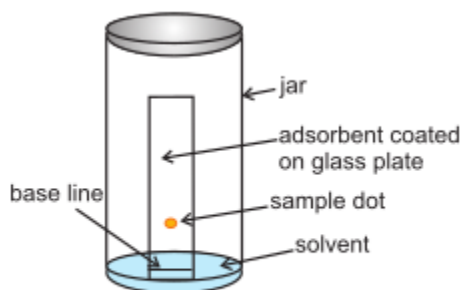


Figure 8(a) Thin layer chromatography

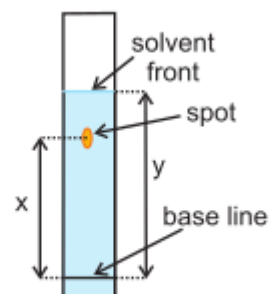


Figure 8(b) Developed chromatogram.

The spots of coloured compounds are visible on TLC plate due to 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 ultraviolet light. Another detection technique is to place the plate in a covered jar containing a few crystals of iodine. Spots of compounds, which adsorb iodine, will show up as brown spots. Sometimes an appropriate reagent may also be sprayed on the plate. For example, amino acids may be detected by spraying the plate with ninhydrin solution (Figure 8b).

2.5.2 Partition Chromatography

Partition chromatography is based on continuous differential partition of components of a mixture between stationary and mobile phases. Paper chromatography is a type of partition chromatography. In paper chromatography, a special quality paper known as chromatography paper is used. Chromatography paper contains water trapped in it, which acts as the stationary phase. A strip of chromatography paper spotted at the base with the solution of the mixture is suspended in a suitable solvent or a mixture of solvents (Figure 9). This solvent acts as the mobile phase. The solvent rises up the paper by capillary action and flows over the spot. The paper selectively retains different components according to their differing partition in the two phases. The paper strip so developed is known as a chromatogram.

The spots of the separated coloured compounds are visible at different heights from the position of initial spot on the chromatogram. The spots of the separated colourless compounds may be

observed either under ultraviolet light or by the use of an appropriate spray reagent as discussed under thin layer chromatography.

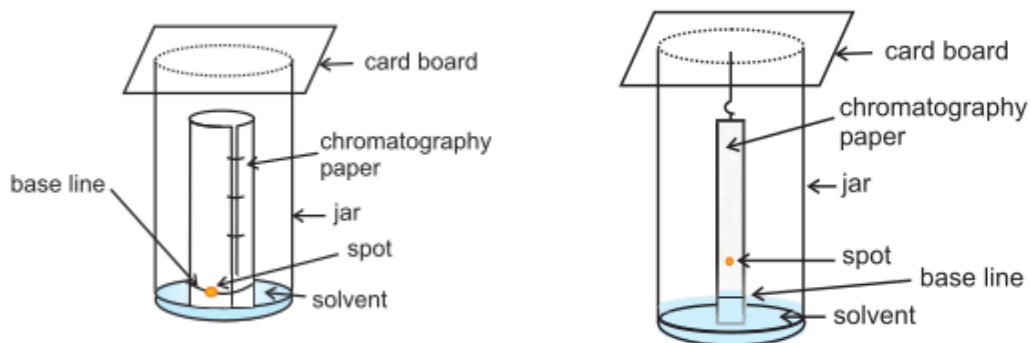


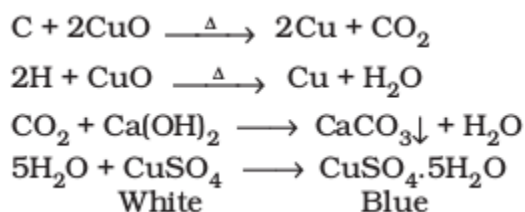
Figure 9 Paper chromatography

3. Qualitative Analysis of Organic Compounds

The elements present in organic compounds are carbon and hydrogen. In addition to these, they may also contain oxygen, nitrogen, sulphur, halogens and phosphorus.

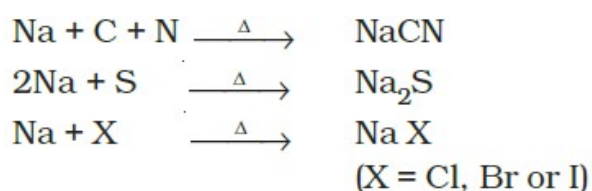
3.1 Detection of Carbon and Hydrogen

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



3.2 Detection of Other Elements

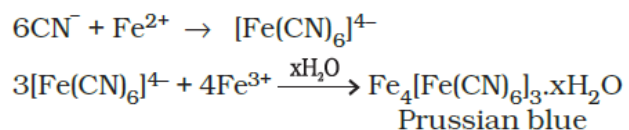
Nitrogen, sulphur, halogens and phosphorus present in an organic compound are detected by Lassaigne's test. The elements present in the compound are converted from covalent form into the ionic form by fusing the compound with sodium metal. Following reactions take place: sulphur from the organic compound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



On sodium fusion C, N, S and X from the organic compound can give cyanide, sulphide and halide of sodium. Then they are extracted from the fused mass by boiling it with distilled water. This extract is known as sodium fusion extract or Lassaigne's extract.

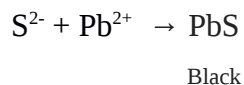
3.2.1 Test for Nitrogen

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

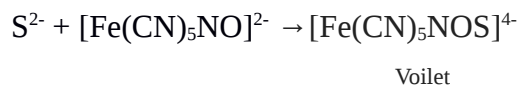


3.2.2 Test for Sulphur

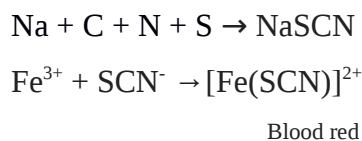
(a) The sodium fusion extract is acidified with acetic acid and lead acetate is added to it. A black precipitate of lead sulphide indicates the presence of sulphur.



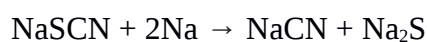
(b) On treating sodium fusion extract with sodium nitroprusside, appearance of a violet colour further indicates the presence of sulphur.



In case, nitrogen and sulphur both are present in an organic compound, sodium thiocyanate is formed. No Prussian blue color is appeared since there are no free cyanide ions, however, It gives blood red colour on treatment with alkali and ferric chloride.

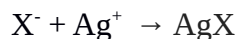


If sodium fusion is carried out with excess of sodium, the thiocyanate decomposes to yield cyanide and sulphide. These ions give their usual tests.



3.2.3 Test for Halogens

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

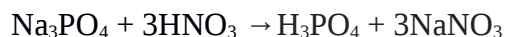


X represents a halogen – Cl, Br or I.

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

3.2.4 Test for Phosphorous

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



Ammonium molybdate

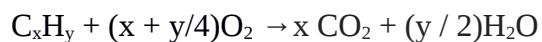
Ammonium phosphomolybdate

4. Quantitative Analysis

Quantitative analysis of compounds is very important in organic chemistry. It helps chemists in the determination of mass percent of elements present in a compound. Mass percent of elements is required for the determination of empirical and molecular formula. The percentage composition of elements present in an organic compound is determined by the following methods:

(1) Carbon and Hydrogen

Both carbon and hydrogen are estimated in one experiment. A known mass of an organic compound is burnt in the presence of excess of oxygen and copper(II) oxide. Carbon and hydrogen in the compound are oxidised to carbon dioxide and water respectively.



The mass of water produced is determined by passing the mixture through a weighed U-tube containing anhydrous calcium chloride. Carbon dioxide is absorbed in another U-tube containing concentrated solution of potassium hydroxide. These tubes are connected in series (**Fig. 9**).

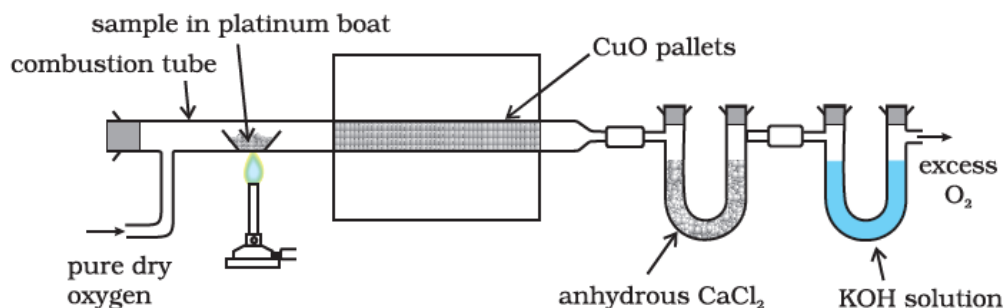


Figure 10. Estimation of carbon and hydrogen

The increase in masses of calcium chloride and potassium hydroxide gives the amounts of water and carbon dioxide from which the percentages of carbon and hydrogen are calculated. Let the mass of organic compound be m g, mass of water and carbon dioxide produced be m_1 and m_2 g respectively;

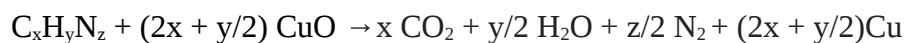
$$\text{Percentage of carbon} = \frac{12 \times m_2 \times 100}{44 \times m}$$

$$\text{Percentage of hydrogen} = \frac{2 \times m_1 \times 100}{18 \times m}$$

(2) Nitrogen

There are two methods for estimation of nitrogen: (i) Dumas method and (ii) Kjeldahl's method.

(i) Dumas method: The nitrogen containing organic compound, when heated with copper oxide in an atmosphere of carbon dioxide, yields free nitrogen in addition to carbon dioxide and water.



Traces of nitrogen oxides formed, if any, are reduced to nitrogen by passing the gaseous mixture over heated copper gauze. The mixture of gases so produced is collected over an aqueous

solution of potassium hydroxide which absorbs carbon dioxide. Nitrogen is collected in the upper part of the graduated tube (**Figure 11**).

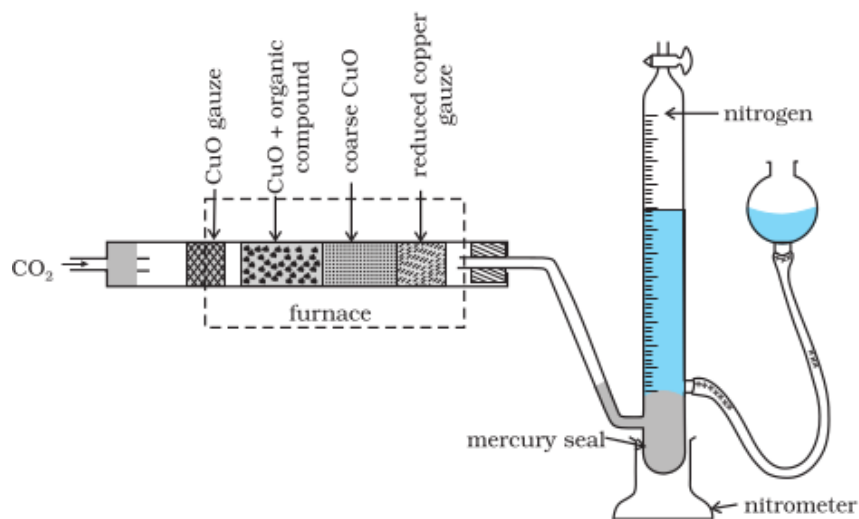


Figure 11. Dumas method

Let the mass of organic compound = m g

Volume of nitrogen collected = V_1 mL

Room temperature = T_1 K

$$\text{Volume of nitrogen at STP} = V = \frac{P_1 V_1 \times 273}{760 \times T_1}$$

Where p_1 and V_1 are the pressure and volume of nitrogen, p_1 is different from the atmospheric pressure at which nitrogen gas is collected. The value of p_1 is obtained by the relation;

$p_1 = \text{Atmospheric pressure} - \text{Aqueous tension}$

22400 mL N_2 at STP weighs 28 g.

$$V \text{ mL } N_2 \text{ at STP weighs} = \frac{28 \times V}{22400} \text{ g}$$

$$\text{Percentage of nitrogen} = \frac{28 \times V \times 100}{22400 \times m}$$

(ii) Kjeldahl's method: In this method, the compound containing nitrogen is heated with concentrated sulphuric acid and nitrogen in the compound gets converted to ammonium sulphate (**Figure 12**). The resulting acid mixture is then heated with excess of sodium hydroxide. The liberated ammonia gas is absorbed in an excess of standard solution of sulphuric acid. The amount of ammonia produced is determined by estimating the amount of sulphuric acid

consumed in the reaction. It is done by estimating unreacted sulphuric acid left after the absorption of ammonia by titrating it with standard alkali solution. The difference between the initial amount of acid taken and that left after the reaction gives the amount of acid reacted with ammonia.

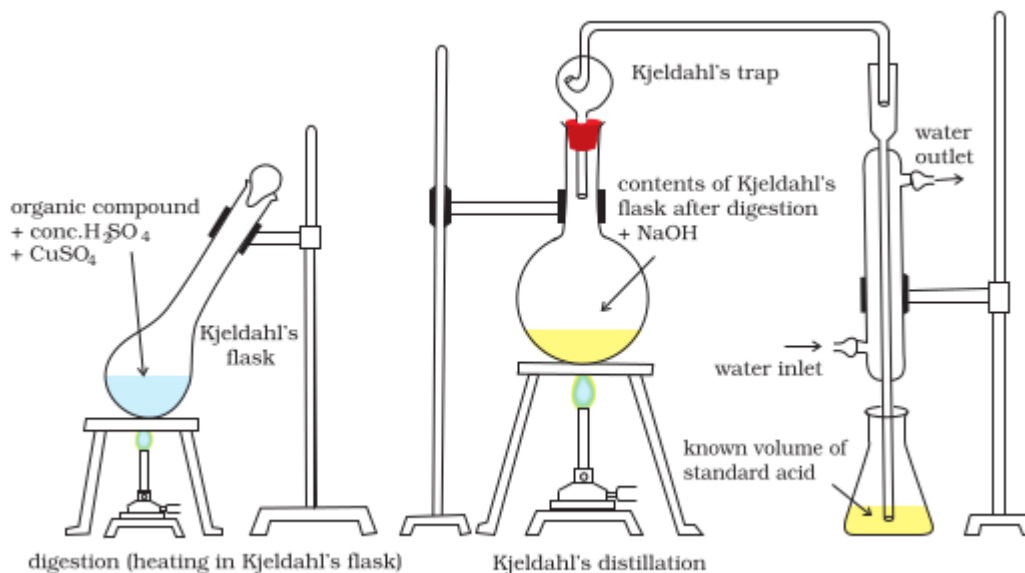
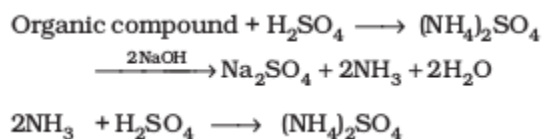


Figure 12. Kjeldahl's method



Let the mass of organic compound taken = m g

Volume of H₂SO₄ of molarity, M, taken = V mL

Volume of NaOH of molarity, M, used for titration of excess of H₂SO₄ = V₁ mL

V₁mL of NaOH of molarity M = V₁/2 mL of H₂SO₄ of molarity M

Volume of H₂SO₄ of molarity M unused = (V - V₁/2) mL

(V - V₁/2) mL of H₂SO₄ of molarity M = 2(V - V₁/2) mL of NH₃ solution of molarity M.

1000 mL of 1 M NH₃ solution contains 17g NH₃ or 14 g of N

2(V - V₁/2) mL of NH₃ solution of molarity M contains Nitrogen

$$= \frac{14 \times M \times 2 \left(V - \frac{V_1}{2} \right)}{1000} \text{ g}$$

$$\text{percentage of } N = \frac{14 \times M \times 2 \left(V - \frac{V_1}{2} \right)}{1000} \times \frac{100}{m}$$

$$= \frac{1.4 \times M \times 2 \left(V - \frac{V_1}{2} \right)}{m}$$

Kjeldahl method is not applicable to compounds containing nitrogen in nitro and azo groups and nitrogen present in the ring (e.g. pyridine) as nitrogen of these compounds does not change to ammonium sulphate under these conditions.

(3) Halogens

Carius method: A known mass 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, (Figure 13) in a furnace. Carbon and hydrogen present in the compound are oxidised to carbon dioxide and water. The halogen present forms the corresponding silver halide (AgX). It is filtered, washed, dried and weighed.

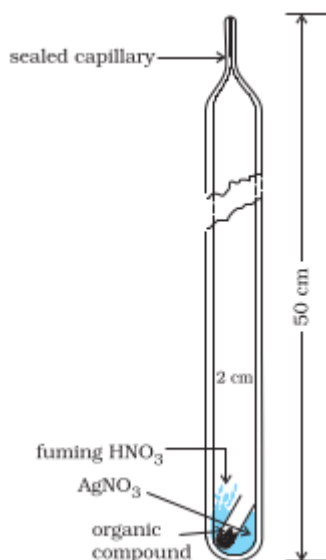


Figure 13. Carius method

Let the mass of organic compound taken = m g

Mass of AgX formed = m_1 g

1 mol of AgX contains 1 mol of X

Mass of halogen in m_1 g of AgX

$$\frac{\text{atomic mass of } X \times m_1}{\text{molecular mass of } AgX} g$$

$$\text{percentage of halogen} = \frac{\text{atomic mass of } X \times m_1 \times 100}{\text{molecular mass of } AgX \times m}$$

(4) Sulphur

A known mass of an organic compound is heated in a Carius tube with sodium peroxide or fuming nitric acid. Sulphur present in the compound is oxidised to sulphuric acid. It is precipitated as barium sulphate by adding excess of barium chloride solution in water. The precipitate is filtered, washed, dried and weighed. The percentage of sulphur can be calculated from the mass of barium sulphate.

Let the mass of organic compound taken = m g and the mass of barium sulphate formed = m_1 g

1 mol of $BaSO_4$ = 233 g $BaSO_4$ = 32 g sulphur

$$m_1 \text{ g } BaSO_4 \text{ contains } \frac{32 \times m_1}{233} \text{ g sulphur}$$

$$\text{percentage of sulphur} = \frac{32 \times m_1 \times 100}{233 \times m}$$

(5) Phosphorus

A known mass of an organic compound is heated with fuming nitric acid whereupon phosphorus present in the compound is oxidised to phosphoric acid. It is precipitated as ammonium phosphomolybdate, $(NH_4)_3PO_4 \cdot 12MoO_3$, by adding ammonia and ammonium molybdate. Alternatively, phosphoric acid may be precipitated as $MgNH_4PO_4$ by adding magnesia mixture which on ignition yields $Mg_2P_2O_7$.

Let the mass of organic compound taken = m g and mass of ammonium phosphomolybdate = m_1 g

Molar mass of $(NH_4)_3PO_4 \cdot 12MoO_3$ = 1877g

$$\text{percentage of phosphorous} = \frac{31 \times m_1 \times 100}{1877 \times m}$$

If phosphorous is estimated as $Mg_2P_2O_7$

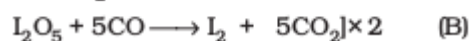
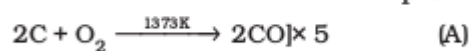
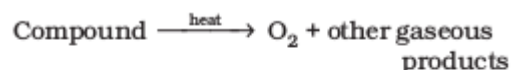
$$\text{percentage of phosphorous} = \frac{62 \times m_1 \times 100}{222 \times m}$$

Where, 222 u is the molar mass of $Mg_2P_2O_7$, m , the mass of organic compound taken, m_1 , the mass of $Mg_2P_2O_7$ formed and 62, the mass of two phosphorus atoms present in the compound $Mg_2P_2O_7$.

(6) Oxygen

The percentage of oxygen in an organic compound is usually found by difference between the total percentage composition (100) and the sum of the percentages of all other elements. However, oxygen can also be estimated directly as follows:

A definite mass of an organic compound 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 (I_2O_5) when carbon monoxide is oxidised to carbon dioxide producing iodine.



On making the amount of CO produced in equation (A) equal to the amount of CO used in equation (B) by multiplying the equations (A) and (B) by 5 and 2 respectively; we find that each mole of oxygen liberated from the compound will produce two moles of carbon dioxide. Thus 88 g carbon dioxide is obtained if 32 g oxygen is liberated.

Let the mass of organic compound taken be m g and mass of carbon dioxide produced be m_1 g

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

$$\text{percentage of oxygen} = \frac{32 \times m_1 \times 100}{88 \times m}$$

The percentage of oxygen can be derived from the amount of iodine produced also.

Presently, the estimation of elements in an organic compound is carried out by using microquantities of substances and automatic experimental techniques. 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 the substance (1-3 mg) and displays the values on a screen within a short time.

5. Summary

- Purification, qualitative and quantitative analysis of organic compounds are carried out for determining their structures.
- The different methods of purification are crystallization, sublimation, distillation and differential extraction.

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- Chromatography is a useful technique of separation, identification and purification of compounds. There are two types of chromatography: adsorption chromatography and partition chromatography.
 - For pure organic compound, qualitative analysis is carried out for detection of elements present in it. However, quantitative analysis is done to estimate the percentage of various elements in the organic compound.