

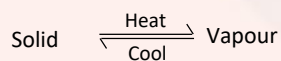
Purification and Characterization of Organic Compounds

Purification of organic compounds: A large number of methods are available for the purification of substances

(i) **Simple crystallisation:** This is the most common method used to purify organic solids. It is based upon the fact that whenever a crystal is formed, it tends to leave out the impurities.

(ii) **Fractional crystallisation:** The process of separation of different components of a mixture by repeated crystallisations is called fractional crystallisation.

(iii) **Sublimation:** Certain organic solids on heating directly change from solid to vapour state without passing through a liquid state, such substances are called *sublimable* and this process is called *sublimation*.



(iv) **Steam distillation:** This method is applicable for the separation and purification of those organic compounds (solids or liquids) which (a) are insoluble in water (b) are

volatile in steam (c) possess a high vapour pressure (10-15 *mm Hg*) at 373 K and (d) contain non-volatile impurities.

(vii) **Azeotropic distillation:** Azeotropic mixture is a mixture having constant boiling point. The most familiar example is a mixture of ethanol and water in the ratio of 95.87 : 4.13 (a ratio present in rectified spirit). It boils at 78.13°C.

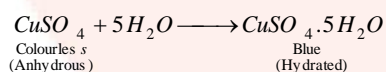
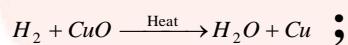
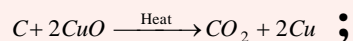
Types of chromatography: Depending upon the nature of the stationary and the mobile phases, the different types of chromatographic techniques commonly used are in a given table,

Type of Chromatography	Mobile/Stationary Phase	Uses
Adsorption or column chromatography	Liquid/Solid	Large scale separations
Thin-layer chromatography	Liquid/Solid	Qualitative analysis (identification and characterization of organic compounds)
High performance liquid	Liquid/Solid	Qualitative and quantitative analysis

chromatography		
Gas-liquid chromatography (GLC)	Gas/Liquid	Qualitative and quantitative analysis
Partition chromatography or ascending paper chromatography	Liquid/Liquid	Qualitative and quantitative analysis of polar organic compounds (sugars, α -amino acids and inorganic compounds)

Qualitative analysis:

Carbon is an essential component of an organic compound. The following method is known as the copper oxide test.



Lassaigne method

This is used to detect nitrogen, halogen and sulphur.

Lassaigne method (Detection of elements)

Element	Sodium Extract (S.E.)	Confirmed Test	Reaction
Nitrogen	$\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}$ (S.E.)	S.E. + $\text{FeSO}_4 + \text{NaOH}$, boil and cool + $\text{FeCl}_3 + \text{conc. HCl}$ Blue or green colour	$2\text{NaCN} + \text{FeSO}_4 \longrightarrow \text{Fe}(\text{CN})_2 + \text{Na}_2\text{SO}_4$ $\text{Fe}(\text{CN})_2 + 4\text{NaCN} \longrightarrow \text{Na}_4[\text{Fe}(\text{CN})_6]$ Sodium ferrocyanide $3\text{Na}_4[\text{Fe}(\text{CN})_6] + 4\text{FeCl}_3 \xrightarrow{\text{HCl}} \text{Fe}_4[\text{Fe}(\text{CN})_6]_3 + 12\text{NaCl}$ Ferric ferrocyanide (Prussian blue)
Sulphur	$2\text{Na} + \text{S} \xrightarrow{\Delta} \text{Na}_2\text{S}$ (S.E.)	(i) S.E. + sodium nitro prusside (ii) S.E. + $\text{CH}_3\text{CO}_2\text{H} + (\text{CH}_3\text{CO}_2)_2\text{Pb}$ A black ppt.	(i) $\text{Na}_2\text{S} + \text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \longrightarrow \text{Na}_4[\text{Fe}(\text{CN})_5\text{NO.S}]$ or Sodium nitroprusside (Purple) $\text{Na}_3[\text{Fe}(\text{ONSNa})(\text{CN})_5]$ Sodium thionitroprusside (Violet) (ii) $\text{Na}_2\text{S} + (\text{CH}_3\text{COO})_2\text{Pb} \xrightarrow{\text{CH}_3\text{COOH}} \text{PbS} \downarrow + 2\text{CH}_3\text{COONa}$ black ppt.
Halogen	$\text{Na} + \text{X} \xrightarrow{\Delta} \text{NaX}$ (S.E.) ($\text{X} = \text{Cl}, \text{Br}, \text{I}$)	S.E. + $\text{HNO}_3 + \text{AgNO}_3$ (i) White ppt soluble in aq NH_3 confirms Cl. (ii) Pale yellow ppt partially soluble in aq. NH_3 confirms Br. (iii) Yellow ppt insoluble in aq NH_3 confirms I.	$\text{NaX} + \text{AgNO}_3 \xrightarrow{\text{HNO}_3} \text{AgX} \downarrow$ ppt $\text{AgCl} + 2\text{NH}_3(\text{aq}) \longrightarrow [\text{Ag}(\text{NH}_3)_2]\text{Cl}$ White ppt soluble $\text{AgBr} + 2\text{NH}_3(\text{aq}) \rightarrow [\text{Ag}(\text{NH}_3)_2]\text{Br}$ Yellow ppt. Partially soluble $\text{AgI} + \text{NH}_3(\text{aq}) \longrightarrow \text{Insoluble}$
Nitrogen and sulphur together	$\text{Na} + \text{C} + \text{N} + \text{S} \xrightarrow{\Delta} \text{NaCNS}$ (S.E.) with excess of Na the thiocyanate formed decomposes into cyanide and sulphide. $\text{NaCNS} + 2\text{Na} \rightarrow \text{NaCN} + \text{Na}_2\text{S}$	As in test for nitrogen; instead of green or blue colour, blood red colouration confirms presence of N and S both.	$3\text{NaCNS} + \text{FeCl}_3 \longrightarrow [\text{Fe}(\text{SCN})_3 \text{ or } [\text{Fe}(\text{SCN})_3]\text{Cl}_2 + 3\text{NaCl}$ Ferric sulphocyanide (Blood red colour)

Calculation of Empirical and Molecular formula

(i) **Empirical formula:** Empirical formula of a substance gives the simplest whole number ratio between the atoms of the various elements present in one molecule of the

substance. For example, empirical formula of glucose is CH_2O , i.e. for each carbon atom, there are two *H*-atoms and one oxygen atom. Its molecular formula is however, $C_6H_{12}O_6$.

(ii) Molecular formula: *Molecular formula of a substance gives the actual number of atoms present in one molecule of the substance.*

Molecular formula = $n \times$ Empirical formula

Where, n is a simple integer 1, 2, 3,..... etc. given by the equation,

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