



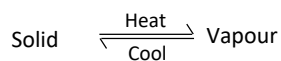
## Purification and Characterization of Organic Compounds

**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,

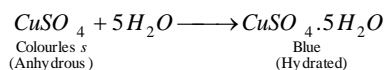
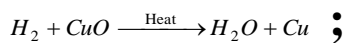
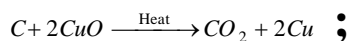
Type of Chromatography	Mobile/Stationary Phase	Uses
Adsorption or column chromatography	Liquid/Solid	Large scale separations
Thin-layer chromatography	Liquid/Solid	Qualitative analysis (identification and characterization of organic compounds)
High performance liquid	Liquid/Solid	Qualitative and quantitative 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, $\alpha$ -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.) (X = Cl, Br, I)	S.E. + $\text{HNO}_3 + \text{AgNO}_3$  (i) White ppt soluble in aq. $\text{NH}_3$ confirms Cl. (ii) Pale yellow ppt partially soluble in aq. $\text{NH}_3$ confirms Br. (iii) Yellow ppt insoluble in aq $\text{NH}_3$ 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  $\text{AgBr} + 2\text{NH}_3(\text{aq}) \longrightarrow [\text{Ag}(\text{NH}_3)_2]\text{Br}$ 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.	$3\text{NaCNS} + \text{FeCl}_3 \longrightarrow [\text{Fe}(\text{SCN})_3] \text{ or } [\text{Fe}(\text{SCN})_2]\text{Cl}_2 + 3\text{NaCl}$ 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,

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