

MIND MAP

POC & Purification

Element	Sodium Extract (S.E.)	Confirmed Test	Reaction
Nitrogen	$\text{Na} + \text{C} + \text{N} \xrightarrow{\Delta} \text{NaCN}_{(\text{S.E.})}$	S.E. + $\text{FeSO}_4 + \text{NaOH}$, boil and cool + FeCl_3 + conc. HCl Blue or green colour	$2\text{NaCN} + \text{FeSO}_4 \rightarrow \text{Fe}(\text{CN})_2 + \text{Na}_2\text{SO}_4$ $\text{Fe}(\text{CN})_2 + 4\text{NaCN} \rightarrow \text{Na}_4[\text{Fe}(\text{CN})_6]$ Sodium ferrocyanide $3\text{Na}_4[\text{Fe}(\text{CN})_6] + 4\text{FeCl}_3 \xrightarrow{\text{HCl}} \text{Fe}[\text{Fe}(\text{CN})_6]_3 + 12\text{NaCl}$ Ferric Ferrocyanide (Prussian blue)
Sulphur	$2\text{Na} + \text{S} \xrightarrow{\Delta} \text{Na}_2\text{S}_{(\text{S.E.})}$	(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) $\text{Na}_2\text{S} + \text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \rightarrow \text{Na}_4[\text{Fe}(\text{CN})_5\text{NOS}]$ 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	$\text{Na} + \text{X} \xrightarrow{\Delta} \text{NaX}_{(\text{S.E.})}$ (X = Cl, Br, 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 $\text{AgCl} + 2\text{NH}_3(\text{aq}) \rightarrow [\text{Ag}(\text{NH}_3)_2]\text{Cl}$ White ppt soluble $\text{AgBr} + 2\text{NH}_3(\text{aq}) \rightarrow [\text{Ag}(\text{NH}_3)_2]\text{Br}$ Yellow ppt Partially soluble $\text{AgI} + \text{NH}_3(\text{aq}) \rightarrow \text{Insoluble}$

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Nitrogen and sulphur together	$\text{Na} + \text{C} + \text{N} + \text{S} \xrightarrow{\Delta} \text{NaCNS}_{(\text{S.E.})}$ 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.	$3\text{NaCNS} + \text{FeCl}_3 \rightarrow [\text{Fe}(\text{SCN})_3] \text{ or } [\text{Fe}(\text{SCN})] \text{Cl}_2 + 3\text{NaCl}$ Ferric sulphocyanide (Blood red colour)

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Nitrogen	<u>Soda lime test</u> : A pinch of an organic compound is heated strongly with soda lime ($\text{NaOH} + \text{CaO}$) in a test tube. If ammonia gas evolves, it indicates nitrogen. $\text{CH}_3\text{CONH}_2 + \text{NaOH} \xrightarrow{\text{CaO}} \text{CH}_3\text{COONa} + \text{NH}_3$. This test is, however, not reliable since certain Acetamide compounds like nitro, azo etc. do not evolve NH_3 when heated with soda lime.
Sulphur	<u>Oxidation test</u> : Sulphur can also be tested by oxidation test. The organic compound is fused with fusion mixture (a mixture of sodium carbonate and potassium nitrate). The sulphur, if present in the organic compound, is oxidised to sodium sulphate. $\text{Na}_2\text{CO}_3 + \text{S} + 3\text{O} \rightarrow \text{Na}_2\text{SO}_4 + \text{CO}_2$. The fused mass is dissolved in water and the solution is acidified with hydrochloric acid. Barium chloride solution is then added. The formation of a white precipitate indicates the presence of sulphur. $\text{Na}_2\text{SO}_4 + \text{BaCl}_2 \rightarrow \text{BaSO}_4 + 2\text{NaCl}$ (White ppt.)
Halogen	<u>Beilstein's test (copper wire test)</u> : A clean copper wire is heated in the Bunsen flame till it does not impart any green colour to the flame. The heated end is dipped in the organic compound and heated again. The appearance of a green or bluish green flame due to the formation of volatile cupric halides indicates the presence of some halogen in the organic compound. Though this test is very sensitive yet it does not confirm the presence of halogens in an organic compound since certain organic compounds like urea, thiourea, pyridine, organic acids etc. which do not contain halogens give this test due to the formation of volatile cupric cyanide. It does not tell as to which halogen is present. <u>Special test for bromine and iodine (layer test)</u> : Boil a portion of the Lassaigne's extract with nitric acid. Add a few drops of CS_2 and then add chlorine water slowly with constant shaking. An orange colouration in CS_2 layer confirms the presence of bromine where as a violet colouration in the layer confirms the presence of iodine. $2\text{NaBr} + \text{Cl}_2 \rightarrow 2\text{NaCl} + \text{Br}_2$; $2\text{NaI} + \text{Cl}_2 \rightarrow 2\text{NaCl}_2 + \text{I}_2$ turns CS_2 layer orange turns CS_2 layer violet

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Phosphorus	Phosphorus is detected by fusing the organic compound with sodium peroxide when phosphorus is converted into sodium phosphate. $2P + 5Na_2O_2 \rightarrow 2Na_3PO_4 + 2Na_2O$. The fused mass is extracted with H_2O, boiled with conc. HNO_3 and then ammonium molybdate is added. Appearance of yellow ppt. or colouration due to the formation of ammonium phosphomolybdate indicates the presence of phosphorus. $Na_3PO_4 + 3HNO_3 \xrightarrow{\Delta} H_3PO_4 + 3NaNO_3$ $H_3PO_4 + 12(NH_4)_2MnO_4 + 21HNO_3 \rightarrow (NH_4)_3PO_4 \cdot 12MnO_3 + 21NH_4NO_3 + 12H_2O$ Amm.molybdate Amm.Phosphomolybdate (yellow ppt.)
Oxygen	There is no satisfactory qualitative method for the detection of oxygen. However, its presence can be inferred indirectly. (i) If the organic compound is heated alone in a dry test tube in the presence of nitrogen, the formation of water drops on cooler parts of the tube may indicate the presence of oxygen. (ii) The presence of oxygen can be inferred by testing the presence of functional groups known to contain oxygen, e.g., hydroxyl ($-OH$), aldehydic ($-CHO$), carboxyl ($-COOH$) groups, etc.

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Element	Method and its principle	Formula
Carbon and Hydrogen	<u>Liebig's combustion method</u> : In this method, a known weight of organic compound is heated with pure and dry cupric oxide in a steam of pure and dry oxygen, when carbon is oxidised to carbon dioxide while hydrogen is oxidised to water. From the weight of CO₂ and H₂O, the percentage of C and H can be calculated. $C_xH_y + \left(x + \frac{y}{4}\right) O_2 \xrightarrow{\Delta} xCO_2 + \frac{y}{2} H_2O$	(i) % of C = $\frac{\text{Weight of CO}_2}{\text{Weight of org. compound}} \times \frac{12}{44} \times 100$ (ii) % of H = $\frac{\text{Weight of H}_2\text{O}}{\text{Weight of org. compound}} \times \frac{2}{18} \times 100$
Nitrogen	(i) Duma's method : Elemental nitrogen is converted into molecular nitrogen by a suitable chemical method and its volume is changed to STP data. $C + 2H + 3CuO \rightarrow CO_2 + H_2O + 3Cu$ $2N + 2CuO \rightarrow N_2 + \text{oxide of nitrogen}$ Oxides of nitrogen + Cu → N₂ + CuO (ii) <u>Kjeldahl's method</u> : Nitrogen in organic compound is converted into NH₃ by suitable chemical method which, in turn, is absorbed by V₁ mL of N₁ H₂S₄. $N(\text{from organic compound}) + \text{conc. H}_2\text{SO}_4 \xrightarrow{\Delta} (NH_4)_2SO_4$ $(NH_4)_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + 2H_2O + 2NH_3$	% of N = $\frac{28}{22400} \times \frac{V}{W} \times 100$ Where, V= volume of N₂ in nitrometer (in ml) at NTP, W = Weight of substance taken; % of N = $\frac{1.4 \times N \times V}{W}$ Note : This method is, however, not applicable to compounds containing nitrogen in the ring (e.g. Pyridine, quinoline etc.) and compounds containing nitro and azo (-N=N-) groups since nitrogen in these compounds is not completely converted into (NH₄)₂SO₄ during digestion.

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Halogens	(i) Carius method: The method is based on the fact that when an organic compound containing halogen (Cl, Br, or I) is heated in a sealed tube with fuming nitric acid in presence of silver nitrate, silver halide is formed. From the mass of silver halide formed, the percentage of the halogen can be calculated.	$\% \text{ of Cl} = \frac{35.5}{143.5} \times \frac{\text{Mass of AgCl formed}}{\text{Mass of substance taken}} \times 100$ $\% \text{ of Br} = \frac{80}{188} \times \frac{\text{Mass of AgBr formed}}{\text{Mass of substance taken}} \times 100$ $\% \text{ of I} = \frac{127}{235} \times \frac{\text{Mass of AgI formed}}{\text{Mass of substance taken}} \times 100$
Sulphur	<u>Carius method:</u> When an organic compound containing sulphur is heated with fuming nitric acid, sulphur is oxidised to sulphuric acid. This is precipitated as barium sulphate by adding barium chloride solution. From the amount of barium sulphate, percentage of sulphur can be calculated. $\text{S} + \text{HNO}_3 (\text{fuming}) \xrightarrow{\text{heat}} \text{H}_2\text{SO}_4$ $\text{H}_2\text{SO}_4 + \text{BaCl}_2 \rightarrow \text{BaSO}_4 + 2\text{HCl}$ white ppt	$\% \text{ of S} = \frac{32}{233} \times \frac{\text{Mass of BaSO}_4 \text{ formed}}{\text{Mass of substance taken}} \times 100$

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phosphorous	<u>Carius method</u>: The organic compound containing phosphorus is heated with fuming nitric acid. Phosphorus is oxidised to phosphoric acid. It is precipitated as magnesium ammonium phosphate, MgNH_4PO_4, by the addition of magnesia mixture ($\text{MgSO}_4 + \text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$). The magnesium ammonium phosphate is washed, dried and ignited when it is converted to magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$). $2\text{MgNH}_4\text{PO}_4 \xrightarrow{\text{heat}} \text{Mg}_2\text{P}_2\text{O}_7 + 2\text{NH}_3 + \text{H}_2\text{O}$ From the mass of magnesium pyro-phosphate, the percentage of phosphorus in the compound can be calculated.	$\% \text{ of P} = \frac{62}{222} \times \frac{\text{Mass of Mg}_2\text{P}_2\text{O}_7 \text{ formed}}{\text{Mass of substance taken}} \times 100$ $\% \text{ P} = \frac{31}{1877} \times \frac{\text{Mass of } [\text{NH}_4]_3\text{PMo}_{12}\text{O}_{40}}{\text{Mass of substance taken}} \times 100$
Oxygen	(i) The usual method of determining the percentage of oxygen in an organic compound is by the method of difference. All the elements except oxygen present in the organic compound are estimated and the total of their percentages subtracted from 100 to get the percentage of oxygen. (ii) <u>Aluise's method</u>: Organic compound containing oxygen is heated with graphite and CO formed is quantitatively converted into CO on reaction with IO. $\text{Org. compound} \xrightarrow{\text{Pyrolysis}} \text{Oxygen}$ $\text{O}_2 + 2\text{C} \xrightarrow{1100^\circ\text{C}} 2\text{CO}$ $5\text{CO} + \text{I}_2\text{O}_5 \rightarrow \text{I}_2 + 5\text{CO}_2$	Percentage of oxygen = 100 – (Sum of the percentages of all other elements) $\text{O} \equiv \text{CO} \equiv \text{CO}_2$ $\% \text{ of O} = \frac{16}{44} \times \frac{\text{mass of CO}_2}{\text{mass of org. compd.}} \times 100$