[B] INORGANIC QUALITATIVE ANALYSIS

Inorganic Qualitative analysis deals with the detection of an unknown powder or solution by the systematic chemical methods. The substance under analysis is generally composed of two parts. Each one is known as radical. Like, positive radical or cation (+Ve) and negative radical or anion (–Ve).

Usually the substance under analysis is subjected to:

(A) Dry tests (For substance in the solid state only).

(B) Wet tests (For substance in the aqueous state only and for the solution prepared from solid salts).

General procedure for the analysis of the solid substances is as follows:

Aim : Qualitative analysis of given unknown Inorganic substance.

(A) Physical properties

(B) Dry tests for positive radicals or cations

(C) Dry tests for negative radicals or anions

(D) Conclusion from dry test :

(a) The probable positive radicals : _____

(b) The probable negative radicals : _____

(E) Preparation of original solution

(F) Wet tests for positive radicals or cations

(G) Separation of cation into the groups (metals into groups)

(H) Insoluble phosphate scheme (Only when substance is water insoluble)

(I) Confirmative test for positive radicals or cations

(J) Wet tests for negative radicals or anions (Only when substance is water soluble)

(K) Confirmative test for negative radicals

(L) Equation for Cation and Anion

(M) Final conclusion:

The given unknown Inorganic substance contains:

(a) Positive radical : _____

(b) Negative radical : _____

[A] PHYSICAL PROPERTIES :

NO.	TEST	OBSERVATION	INFERENCE
1	State	Crystalline (sandy powder)	∴ All soluble substances like K^+, NH_4^+, Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-} etc. may be present.
		Amorphous (Soft powder)	∴ Insoluble substances generally CO_3^{2-}, S^{2-}, PO_4^{3-}, O^{2-} etc. may be present.
2	Colour	White	∴ Generally compounds of Al^{3+}, Zn^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+}, K^+, NH_4^+ etc. may be present.
		Black or Brown	∴ MnO_2, CuO, Ni_2O, CdO, CuS, FeS, FeO, Fe_2O_3 and some sulphides like SnS, FeS etc. may be present.
		Green	∴ Cr^{3+}, Fe^{2+}(ous), Ni^{2+}, $CuCl_2$, $FeSO_4$ may be present.
		Blue	∴ Cu^{2+}, $Co_3(PO_4)_2$ may be present.
		Yellow	∴ CrO_4^{-2}, CdS, PbO, Bi_2O_3, sulphides of Cd^{2+}, $FePO_4$, $FeCl_3$ (Reddish yellow) may be present.
		Orange	∴ $Cr_2O_7^{2-}$, Sb_2O_3 may be present.
		Light pink	∴ $MnCl_2$ or $MnSO_4$ may be present.
		Red (Dark Pink)	∴ Co^{2+}(hydrated), Pb_3O_4, Sb_2S_3 etc. may be present.
		Reddish Brown	∴ Fe_2CO_3 may be present.
3	Odour (smell)	Ammonical smell	∴ $(NH_4)_2CO_3$, NH_4OH, Salts of NH_4^+ may be present.
		Rotten eggs (H_2S)	∴ Soluble sulphides like K_2S, $(NH_4)_2S$ and insoluble sulphodes like ZnS, CuS etc. may be present.
4	Solubility in water Substance + H_2O (shake well) Heat if necessary	Soluble	∴ All soluble substances like K^+, NH_4^+, Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-} etc. may be present.
		Insoluble	∴ Insoluble substance like CO_3^{2-}, S^{2-}, PO_4^{3-}, O^{2-} etc. may be present.

5	Action on Litmus paper	Acidic (Blue to red)	∴ Salt of strong acid & weak base or free acid may be present.
		Basic (Red to blue)	∴ Salt of strong base & weak acid or free alkali may be present.
		Neutral (no effect)	∴ Salt of strong acid & strong base or Salt of weak acid & weak base may be present.

[B] DRY TEST FOR POSITIVE RADICAL :

1	**Heating in a dry test tube** **[N.B.]** (Take substance in a dry test tube and heat it.) *Test tube must be dry and before starting heating, bring wet* ***litmus papers***, ***starch paper***, ***dichromate paper***, ***charred matchstic*** *etc.*	Substance melts	∴ Salt of K^+, some salts containing water of crystallization may be present.
		Substance produce cracking noise (Decrepitates)	∴ Some nitrates like $Pb(NO_3)_2$ & crystalline salts like KBr, KI may be present.
		Water condensing on the cooler part of the test tube	∴ Crystalline salt or some hydroxide may be present or hydroscopic like $MgCl_2$ may be present.
		Sublimation takes place on the cooler part of the test tube. **[Perform mirror test]**	∴ Hg^{2+}, Sb^{+3}, As^{+3}, NH_4^+ etc. may be present.
		Change of colour	
		Blackening at high temperature	∴ Cu^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+} etc. may be present.
		Yellow when hot and white when cold	∴ ZnO and many Zn^{2+} salt may be present.
		Yellowish brown	∴ Bi_2O_2 and SnO_2 may be present
		Yellow	∴ PbO and some Pb^{2+} salt may be present.
		Brown	∴ Fe^{3+}, CdO & Cd^{2+} salts may be present.
		Green	∴ Cr^{3+}, CrO_4^{2-}, $Cr_2O_7^{2-}$ etc. may be present.
		No change i.e. white remains white	∴ Al^{3+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+} etc. may be present.
		Evolution of a gas	
		Evolution of reddish brown gas turns to wet starch paper yellow	∴ Br^- may be present.

		Evolution of reddish brown gas no effect on wet starch paper	∴ NO_3^- may be present.
		Evolution of colourless or greenish yellow gas having pungent smell and bleaches (decolourized) wet litmus paper (Blue/red litmus paper turns to white)	∴ Cl^- may be present.
		Evolution of NH_3 gas recognized by smell, alkaline action on litmus, turmeric paper turns to brown and gives a white fumes with a drop of conc. HCl	∴ NH_4^+ may be present.
		Evolution of Violet gas turns to wet starch paper blue	∴ I^- may be present.
		Evolution of a bad smelling gas (rotten eggs) turns to lead acetate paper black or silvery white	∴ S^{2-} may be present. (N.B. Substance must be insoluble in water)
		White fumes of SO_2 gas acidic to litmus and having pungent smell	∴ SO_4^{2-} (hydrated) may be present.
		If the substance is yellow or orange and gives sparks on heating and becomes dark green on cooling.	∴ CrO_4^{2-}, $Cr_2O_7^{2-}$ may be present.
2	**Mirror test** (Perform when white sublimation take place) Substance + Na_2CO_3 + Charcoal dust (Mix. thoroughly & heat the mixture in a dry test tube)	White mirror	∴ Hg^{2+} may be present.
		Orange sublimation	∴ Sb^{3+} may be present.
		Garlic smell	∴ As^{3+} may be present.
		Evolution of NH_3 gas recognized by smell, alkaline action on litmus, turmeric paper turns to brown and gives a white fumes with a drop of conc. HCl	∴ NH_4^+ may be present.
3	**Charcoal cavity test** Make a cavity in the charcoal & moisten it with a drop of water. Put the mixture of Substance + Na_2CO_3 + Charcoal dust (mix well separately) in the cavity. Again put a drop of water on the mixture and heat it with blow pipe in the reducing	Deflagration (Burning of charcoal)	∴ NO_3^-, Peroxide like BaO_2, CrO_4^{2-} may be present.
		Substance fuses and sinks in charcoal	∴ K^+ may be present.
		Sublimation	∴ NH_4^+ may be present.
		White infusible mass which glows when heated **[PERFORM $Co(NO_3)_2$ TEST]**	∴ Ca^{2+}, Sr^{2+}, Ba^{2+}, Zn^{2+}, Al^{3+}, Mg^{2+} etc. may be present.
		Coloured infusible mass which	∴ Cu^{2+}, Mn^{2+}, Fe^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+},

	flame (yellow) for 8-10 minutes.	glows when heated **[PERFORM BOREX BEAD TEST]**	Cr^{3+}, CrO_4^{2-}, $Cr_2O_7^{2-}$ etc. may be present.
		Metallic Beads	
		White soft bead which marking on paper and yellow incrustation	∴ Pb^{2+} may be present.
		Pinkish white bead with yellow incrustation	∴ Bi^{2+} may be present.
		White bead not marking on paper	∴ Sb^{3+} may be present.
		Red scales	∴ Cu^{2+} may be present.
		Brown incrustation	∴ Cd^{2+} may be present.
		No bead, yellow when hot white when cold	∴ Zn^{2+} may be present.
4	**Cobalt nitrate test** Put one or two drops of $Co(NO_3)_2$ solution on a white infusible mass obtained in charcoal cavity and heat with blowpipe in reducing flame. [This test should be performe only when white infusible mass is obtained in charcoal cavity test]	Green mass	∴ Zn^{2+} may be present.
		Blue mass	∴ Al^{3+} or PO_4^{3-} may be present.
		Pink mass	∴ Mg^{2+} may be present.

5 **Borax Bead Test:** Perform this test only when the substance is coloured and leaves a coloured infusible mass in charcoal cavity test.

(i.e. when any one of Cu^{2+}, Cr^{3+}, CrO_4^{2-}, $Cr_2O_7^{2-}$, Co^{2+}, Fe^{2+}, Fe^{3+}, Mn^{2+}, Ni^{2+} is suspected to be present).

Observation		**Inference**
Colour of the bead in the oxidizing flame	**Colour of the bead in the reducing flame**	**Radicals**
dark yellow when hot Green when cold	Green (in hot & cold)	∴ Cr^{3+}, CrO_4^{2-}, $Cr_2O_7^{2-}$ etc. may be present.
Blue (in hot & cold)	Blue (in hot & cold)	∴ Co^{2+} may be present.
Green when hot blue when cold	Colourless when hot Red or opaque when cold	∴ Cu^{2+} may be present.
Yellow	Light green	∴ Fe^{2+}, Fe^{3+} may be preset.
Violet (Amethyst)	Colourless	∴ Mn^{2+} may be present.
Reddish brown when cold	Grey when cold	∴ Ni^{2+} may be present.

<table>
<tr><td>6</td><td colspan="3">Flame Test : Omit this test if Cu^{2+}, Pb^{2+}, Sb^{3+}, Bi^{3+} are detected from the preliminary dry test as the platinum wire is corroded by this radical. This test can however be performed by using a charred matchstick and when a green or bluish green flame is observed then the above metals are present.</td></tr>
<tr><td></td><td>Test</td><td colspan="2">Observation</td><td>Inference</td></tr>
<tr><td rowspan="7"></td><td rowspan="7">Prepare a paste of the substance in a minimum quantity of conc. HCl and perform flame test in oxidizing flame (blue flame).</td><td>Colour of the flame without cobalt glass.</td><td>Colour of the flame through the cobalt glass</td><td>Radicals</td></tr>
<tr><td>Brick red</td><td>Colourless</td><td>∴ Ca^{2+} may be present.</td></tr>
<tr><td>Crimson</td><td>Crimson</td><td>∴ Sr^{2+} may be present.</td></tr>
<tr><td>Light Green</td><td>Green</td><td>∴ Ba^{2+} may be present.</td></tr>
<tr><td>Violet (Pink)</td><td>Crimson</td><td>∴ K^{+} may be present.</td></tr>
<tr><td>Blue or greenish blue</td><td>–</td><td>∴ Cu^{2+} may be present.</td></tr>
<tr><td>Bluish white (Pigeon Coloured)</td><td>–</td><td>∴ Pb^{2+}, Sb^{3+}, Bi^{2+} etc. may be present.</td></tr>
<tr><td>7</td><td>Test for NH_4^+ :
(Sodium Hydroxide Test)
Substance + NaOH (heat)</td><td colspan="2">Evolution of NH_3 gas recognized by smell, alkaline action on litmus, turmeric paper turns to brown and gives a white fumes with a drop of conc. HCl</td><td>∴ NH_4^+ may be present.</td></tr>
</table>

[C] DRY TEST FOR NEGATIVE RADICAL :

N.B. If given substance is insoluble in water then CO_3^{2-}, S^{2-} , O^{2-} and PO_4^{3-} (except salts of NH_4^+ & K^+) are to be present.

1	**Test for CO_3^{2-} and S^{2-} :** Substance + Dilute HCl (if substance is soluble in water and CO_3^{2-} found to be present then K^+ or NH_4^+ is present)	Effervences of CO_2 gas	∴ CO_3^{2-} may be present.
			∴
2	Substance + Dilute HCl (heat)	Evolution of a bad smelling gas (rotten eggs) turns to lead acetate paper black or silvery white	∴ S^{2-} may be present.
3	**Test for Cl^-, Br^-, I^- :** Substance + MnO_2 (powder) + Conc. H_2SO_4 (heat) [**N.B.** Test tube must be dry]	Evolution of colourless or greenish yellow gas having pungent smell and bleaches (decolourized) wet litmus paper (Blue/red litmus paper turns to white)	∴ Cl^- may be present.
		Evolution of reddish brown gas turns to wet starch paper yellow	∴ Br^- may be present.
		Evolution of Violet gas turns to wet starch paper blue	∴ I^- may be present.

4	**Test for NO_3^- :** (Perform when Br^- or I^- found to be absent) Substance + Conc. H_2SO_4 + Cu foil (heat)	Evolution of Reddish brown gas & no effect on wet starch paper	∴ NO_3^- may be present.
5	**Test for PO_4^{3-} :** Substance + few drops of conc. HNO_3 + $(NH_4)_2MoO_4$ in excess (Ammonium molybdate solution) warm slowly	Canary yellow precipitates	∴ PO_4^{3-} may be present.
6	**Test for CrO_4^{2-} :** If given Substance is yellow, add dil. HCl to its aqueous solution	Orange solution	∴ CrO_4^{2-} may be present.
7	**Test for $Cr_2O_7^{2-}$:** If given Substance is orange, add dilute NaOH solution to its aqueous solution	Yellow solution	∴ $Cr_2O_7^{2-}$ may be present.
8	**Test for SO_4^{-2} :** Crystalline substance + H_2O	Soluble	∴ SO_4^{2-} may be present.
9	**Test for O^{-2} :**	All the above radicals are found to be absent and given substance is an amorphous and insoluble in water	∴ O^{2-} may be present.

Note : Sulphates of Lead, Calcium, barium and strontium are sparingly soluble in water.

[D] CONCLUSION FROM DRY TEST :

[i] Probable positive radical : ----------

[ii] Probable negative radical : ----------

[E] PREPARATION OF ORIGINAL SOLUTION FOR WET TEST :

1	Substance + Distilled H_2O (Shake well) (warm if it remains insoluble)	Soluble	∴ O.S. (Possibility of K^+, NH_4^+, NO_3^-, SO_4^{2-}, Cl , Br , I) Or soluble PO_4^{3-} solt like $(NH_4)HPO_4$ or K_3PO_4)
2	Substance + dil. HCl (heat if it is insoluble in cold)	Soluble	∴ O.S. (Possibility of CO_3^{2-}, S^{2-}, PO_4^{3-})
3	Substance + Conc. HCl try to dissolve as much of substance as possible, decant the solution and diluted with distill water.	Soluble	∴ O.S. (Possibility of CO_3^{2-}, S^{2-}, PO_4^{3-})

4	Substance + HNO_3 (heat) (prepared only when 1st group is present)	Soluble	∴ O.S. (Use for 1st group radical)

N.B.

- *If insoluble sulphide (S^{2-}) is present (from dry test), boil the substance with conc. HCl for five minutes to expel H_2S gas. Then check with lead acetate paper till no effect on lead acetate paper i.e. all sulphide (H_2S gas) is expel out.*
- ***Lead acetate paper :** Take a strip of filter paper and dip it into solution of lead acetate.*
- *If the powder (salt) is insoluble in water and **PO_4^{3-}** is detected then follow the **"insoluble phosphate scheme"** (**Page no. 18**) for the identification of the metallic radical after the 1st and 2nd groups.*
- *Use the solution (O.S.) prepared in acid for the determination of only positive radical.*
- *If O.S. is prepared in HCl, then 1st group is absent.*
- *If the substance is soluble in water and phosphate is detected then phosphate of K^+, NH_4^+ may be present*

[F] WET TEST FOR POSITIVE RADICALS :

Classification of positive radical into groups.

1	O.S.+ dilute HCl	White precipitates	∴ I[st] group is present. i.e. Ag^+, Hg^+, Pb^{2+} is present.
2	O.S.+ dil. HCl + H_2S solution (excess) + H_2O (shake well)	Coloured precipitates	∴ II[nd] group is present.
		Black or brown precipitates	∴ Cu^{2+}, Pb^{2+}, Bi^{3+} is present.
		Orange precipitates	∴ Sb^{3+} is present.
		Yellow precipitates	∴ Cd^{2+} is present.
	Test for PO_4^{3-} *: Perform phosphate test* *If phosphate is found to be present and substance is insoluble in water and 1st and 2nd group are absent then follow insoluble phosphate scheme (**page no. 18**) for rest of the elements.*		
	Test for PO_4^{3-} Substance + few drops of conc. HNO_3 + $(NH_4)_2MoO_4$ in excess (Ammonium molybdate solution) warm slowly.	Canary yellow precipitates	∴ PO_4^{3-} is present.
	[*Now proceed as under in **absence of PO_4^{3-}***]		
3	O.S. + NH_4Cl (excess in solid form) + 50% NH_4OH till solution became alkaline	White or coloured precipitates	∴ III[rd] A group is present. i.e. Al^{3+},Fe^{3+},Fe^{2+},Cr^{3+} present.
		White gelatinous sticky precipitates	∴ Al^{3+} is present.

		Reddish brown precipitates	∴ Fe^{3+}(Ferric) is present.
		Green precipitates	∴ Fe^{2+}(Ferrous) or Cr^{3+} is present.
4	O.S. + NH_4Cl (excess in solid form) + 50% NH_4OH + H_2S solution (excess)	White, Buff or Black precipitates	∴ III[rd] B group is present, i.e. Mn^{2+}, Zn^{2+}, Ni^{2+}, Co^{2+} present.
		Buff precipitates Soluble in dilute acetic acid	∴ Mn^{2+} is present.
		White precipitates Insoluble in dil. CH_3COOH	∴ Zn^{2+} is present.
		Black precipitates	∴ Ni^{2+} or Co^{2+} is present.
		O.S. is pink colour.	∴ Co^{2+} is present.
		O.S. is green colour.	∴ Ni^{2+} is present.
5	O.S. + NH_4Cl (Solid) + 50% NH_4OH + saturated solution of $(NH_4)_2CO_3$ excess.	White precipitates	∴ IV[th] Group is present. i.e. Ca^{2+},Sr^{2+}, Ba^{2+} is present.
6	O.S. + NH_4Cl (solid) + 50% NH_4OH + Na_2HPO_4	White precipitates	∴ V[th] A Group is present. i.e. Mg^{2+} is present.
7	**Test for V[th] B Group :**	All the above groups (i.e. from group I to V[th] A) are found to be absent and also given substance is soluble in water	∴ V[th] B Group is present. i.e. K^+, NH_4^+ is present.

[G] SEPARATION OF CATION INTO GROUPS :

GROUP I :

Separation of Ag^+, Hg^+ & Pb^{2+}

1	O.S. + dil. HCl	White precipitates	∴ Ag^+, Hg^{+2} & Pb^{2+} is present.
2	Take small quantity of white precipitates of above test + NH_4OH (excess)	White precipitates unaffected	∴ Pb^{2+} is present.
		White precipitates turns to black	∴ Hg^{+2} is present.
		White precipitates Soluble	∴ Ag^+ is present.

C.T. for Pb^{+2}

1	O.S. + K_2CrO_4	Yellow precipitates of ($PbCrO_4$) soluble in hot NaOH	∴ Pb^{+2} is confirmed.
2	O.S. + KI solution	Yellow precipitates of (PbI_2) soluble in hot (boiling) water, recrystallises into golden yellow needle (plates) on cooling	∴ Pb^{+2} is confirmed.

3	O.S. + NaOH solution	White precipitates of $Pb(OH)_2$ soluble in excess of NaOH	∴ Pb^{+2} is confirmed.

GROUP II :

Separation of Cu^{2+}, Pb^{2+}, Bi^{3+} (Black precipitation)

1	O.S. + NaOH solution	Bluish precipitates turns to black on heating	∴ Cu^{2+} is present.
		White precipitates insoluble in excess of NaOH	∴ Bi^{3+} is present.
		White precipitates soluble in excess of NaOH	∴ Pb^{2+} is present.
2	O.S. + dilute H_2SO_4	White precipitates	∴ Pb^{2+} is present

Separation of As^{3+}, Cd^{2+}, Sb^{3+} (Yellow precipitation)

1	O.S. + dil. HCl + H_2S solution (excess) + H_2O (excess) shake well	Yellow precipitates soluble in conc. HCl (shake well)	∴ Cd^{2+} is present.
		Orange precipitation	∴ Sb^{+3} is present.

C.T. for Cu^{2+}

1	O.S. + NaOH	Blue precipitates turns to black on heating insoluble in excess of NaOH	∴ Cu^{2+} is confirmed.
2	O.S. + NH_4OH	Bluish precipitates Soluble in excess of NH_4OH giving deep blue solution.	∴ Cu^{2+} is confirmed.
3	O.S. + KI	Dirty green precipitates of (Cu_2I_2) appearing brown due to liberation of I_2	∴ Cu^{2+} is confirmed.
4	O.S. + Potassium Ferrocyanide [$K_4Fe(CN)_6$]	Reddish brown precipitates soluble in NH_4OH and insoluble in dilute acid	∴ Cu^{2+} is confirmed.

C.T. for Bi^{3+}

1	O.S. + NaOH	White precipitates of [$Bi(OH)_3$] insoluble in excess of NaOH	∴ Bi^{3+} is confirmed.
2	O.S. + KI	Brown precipitates of (BiI_3) turns to orange solution on addition of excess of KI	∴ Bi^{3+} is confirmed.
3	O.S. + H_2S solution excess	Brown precipitates of (Bi_2S_3) insoluble in tartaric acid (cold dilute)	∴ Bi^{3+} is confirmed.

C.T. for Pb^{+2}

1	O.S. + K_2CrO_4	Yellow precipitates of ($PbCrO_4$) soluble in hot NaOH	∴ Pb^{+2} is confirmed.
2	O.S. + KI	Yellow precipitates of (PbI_2) soluble in hot water, recrystallises into golden yellow needle on cooling	∴ Pb^{+2} is confirmed.
3	O.S. + NaOH	White precipitates of $Pb(OH)_2$ soluble in excess of NaOH	∴ Pb^{+2} is confirmed.
4	O.S. + dil. H_2SO_4	White precipitates	∴ Pb^{+2} is confirmed.

C.T. for Cd^{2+}

1	O.S. + Na_2CO_3	White precipitates of $CdCO_3$	∴ Cd^{2+} is confirmed.
2	O.S. + NaOH	White precipitates of [$Cd(OH)_2$] insoluble in excess of NaOH	∴ Cd^{2+} is confirmed.
3	O.S. + $HgCl_2$	White precipitates of (Hg_2Cl_2) turns to gray in excess of $HgCl_2$	∴ Cd^{2+} is confirmed.

C.T. for Sb^{3+}

1	O.S. + H_2O	White precipitates of (SbOCl) soluble in tartaric acid	∴ Sb^{3+} is confirmed.
2	O.S. + $AgNO_3$ + NaOH (excess)	Black precipitates insoluble in ammonia	∴ Sb^{3+} is confirmed.
3	O.S. + NaOH	White precipitates of Sb_2O_3 soluble in excess of NaOH	∴ Sb^{3+} is confirmed.

GROUP III A :

Separation of Al^{3+}, Fe^{3+}, Fe^{2+} and Cr^{3+}

1	O.S. + NH_4Cl + 50% NH_4OH (excess)	White gelatinous precipitates (O.S. is colourless)	∴ Al^{3+} is present.
		Reddish brown precipitates (O.S. is yellow)	∴ Fe^{3+} (Ferric) is present.
		Dirty green precipitates turns to brown on surface due to exposure in air	∴ Fe^{2+} (Ferrous) is present.
		Bluish green precipitates (O.S. is bluish green)	∴ Cr^{3+} is present.

Separation of Fe^{2+} from Cr^{3+}

1	O.S. + 2-3 drops of Conc. HNO_3 boil and cool + 50 % NH_4OH (excess)	Reddish brown precipitates	∴ Fe^{2+} is present.
		Bluish green precipitates	∴ Cr^{3+} is present.

C.T. for Al^{3+}

1	O.S. + NaOH (shake well)	White gelatinous sticky precipitates soluble in excess of NaOH and reprecipitated by NH_4Cl	∴ Al^{3+} is confirmed.
2	O.S. + NH_4OH + H_2S solution	White precipitates of $Al(OH)_3$ soluble in excess	∴ Al^{3+} is confirmed.
3	O.S. + $K_4Fe(CN)_6$	No precipitates	∴ Al^{3+} is confirmed.

C.T. for Fe^{2+}

1	O.S. + $K_4[Fe(CN)_6]$ (Potassium ferrocyanide)	Bluish white precipitates of $Fe_2[Fe(CN)_6]$	∴ Fe^{2+} is confirmed.
2	O.S. + $K_3[Fe(CN)_6]$ (Potassium ferricyanide)	Dark blue precipitates of $Fe_3[Fe(CN)_6]_2$	∴ Fe^{2+} is confirmed.
3	O.S. + NaOH or NH_4OH	Dirty green precipitates of $Fe(OH)_2$ turns to reddish brown on exposure in air	∴ Fe^{2+} is confirmed.
4	O.S. + Dilute H_2SO_4 + dilute $KMnO_4$ (Two drops) (shake well)	Colour of $KMnO_4$ discharged (disappear)	∴ Fe^{2+} is confirmed.

C.T. for Fe^{3+}

1	O.S. + $K_4Fe(CN)_6$ (Potassium ferrocyanide)	Deep blue precipitates of $Fe_4[Fe(CN)_6]_3$	∴ Fe^{3+} is confirmed.
2	O.S. + $K_3Fe(CN)_6$ (Potassium ferricyanide)	Brown colour solution	∴ Fe^{3+} is confirmed.
3	O.S. + KCNS	Blood red colour $K_3[Fe(SCN)_6]$	∴ Fe^{3+} is confirmed.
4	O.S. + Na_2HPO_4	Yellowish white precipitates of $FePO_4$ insoluble in dilute acetic acid.	∴ Fe^{3+} is confirmed.
5	O.S. + NaOH or NH_4OH	Reddish brown precipitates of $Fe(OH)_3$ insoluble in excess	∴ Fe^{3+} is confirmed.

C.T. for Cr^{3+}

1	O.S. + PbO_2 + NaOH (boil) + acetic acid	Yellow precipitates of $PbCrO_4$	∴ Cr^{3+} is confirmed.
2	O.S. + NH_4OH	Bluish green precipitates of $Cr(OH)_3$ soluble in excess of NH_4OH	∴ Cr^{3+} is confirmed.
3	O.S. + NaOH	Bluish green precipitates of $Cr(OH)_3$	∴ Cr^{3+} is confirmed.

GROUP III B :

Separation of Zn^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}

1	O.S. + NH_4Cl (excess) + 50% NH_4OH + H_2S solution (excess)	White precipitates insoluble in acetic acid	∴ Zn^{2+} is present.
		Pinkish or white or buff coloured precipitates soluble in acetic acid	∴ Mn^{2+} is present.
		Black precipitates	∴ Ni^{2+} or Co^{2+} is present.
2	O.S. + NaOH (shake well)	White precipitates soluble in excess of NaOH	∴ Zn^{2+} is present.
		White precipitates insoluble in excess NaOH	∴ Mn^{2+} is present.
		Light green precipitates insoluble in excess of NaOH (O.S. must be green)	∴ Ni^{2+} is present.
		Blue precipitates turns to pink on heating (O.S. must be pink)	∴ Co^{2+} is present.

C.T. for Zn^{2+}

1	O.S. + $K_4Fe(CN)_6$ (Potassium ferrocyanide)	White precipitates of $Zn_2[Fe(CN)_6]$ insoluble in dil. HCl	∴ Zn^{2+} is confirmed.
2	O.S. + NaOH or NH_4OH	White precipitates of $Zn(OH)_2$ soluble in excess	∴ Zn^{2+} is confirmed.
3	O.S. + $K_3Fe(CN)_6$ (Potassium ferricyanide)	Orange precipitates soluble in dil. HCl	∴ Zn^{2+} is confirmed.

C.T. for Mn^{2+}

1	O.S. + $K_4Fe(CN)_6$ (Potassium ferrocyanide)	Pinkish white precipitates of $Mn_2[Fe(CN)_6]$ soluble in dil. HCl (no precipitation if O.S. is acidic)	∴ Mn^{2+} is confirmed.
2	O.S. + PbO_2 + conc. HNO_3 boil , cool and dilute with water and allow it to settle.	Pink colour of $KMnO_4$	∴ Mn^{2+} is confirmed.

C.T. for Co^{2+}

1	O.S. + sodium nitrite ($NaNO_2$) + CH_3COOH + KI	Yellow precipitates of $[K_3(Co(NO_2)_6)]$	∴ Co^{2+} is confirmed.
2	O.S. + Na_2HPO_4	Violet precipitates of $[Co_3(Po_4)_2]$	∴ Co^{2+} is confirmed.
3	O.S. + NaOH	Blue precipitates of $[Co(OH)_2]$ insoluble in excess of NaOH & turns to pink on heating	∴ Co^{2+} is confirmed.

C.T. for Ni^{2+}

1	O.S. + Br_2 water + NaOH in excess	Black precipitates of ($Ni_2O_4.3H_2O$)	∴ Ni^{2+} is confirmed.
2	O.S. + Dimethyl glyoxime ($C_4H_7C_2N_2$) + 50% NH_4OH excess (shake well)	Deep red precipitates	∴ Ni^{2+} is confirmed.
3	O.S. + NH_4OH (2-3 drops)	Light green precipitates soluble in excess of NH_4OH	∴ Ni^{2+} is confirmed.

GROUP IV :

Separation of Ca^{2+}, Ba^{2+} & Sr^{2+}

1	O.S. + $CaSO_4$ solution (O.S. must be concentrate)	White precipitates immediately	∴ Ba^{2+} is present.
		White precipitates formed on warming or appearing slowly on standing	∴ Sr^{2+} is present.
		No precipitates	∴ Ca^{2+} is present.

C.T. for Ca^{2+}

1	O.S. + K_2CrO_4	No precipitates	∴ Ca^{2+} is confirmed.
2	O.S. + Ammonium oxalate $[(NH_4)_2C_2O_4]$	White precipitates of CaC_2O_4 insoluble in CH_3COOH	∴ Ca^{2+} is confirmed.
3	O.S. + dilute H_2SO_4	White precipitates soluble in ammonium sulphate	∴ Ca^{2+} is confirmed.

C.T. for Ba^{2+}

1	O.S. + K_2CrO_4	Yellow precipitates of ($BaCrO_4$) insoluble in dilute CH_3COOH but soluble in dil. HCl	∴ Ba^{2+} is confirmed.
2	O.S. + Ammonium oxalate $[(NH_4)_2C_2O_4]$	White precipitates of BaC_2O_4 soluble in CH_3COOH on heating	∴ $Ba^{2+}$is confirmed.
3	O.S. + dil. H_2SO_4	White precipitates soluble in all acids like HCl, HNO_3 etc.	∴ Ba^{2+} is confirmed.

C.T. for Sr^{2+}

1	O.S. + K_2CrO_4	Yellow precipitates of ($SrCrO_4$) on heating soluble in CH_3COOH	∴ Sr^{2+} is confirmed.
2	O.S. + $(NH_4)_2C_2O_4$	White precipitates of (SrC_2O_4) soluble in CH_3COOH on heating	∴ Sr^{2+} is confirmed.
3	O.S. + dil. H_2SO_4	White precipitates of (SrC_2O_4) after few minutes	∴ Sr^{2+} is confirmed.

GROUP V A :

Identification of Mg^{+2}

1	O.S. + NH_4Cl + NH_4OH + Na_2HPO_4	White precipitates	∴ Mg^{2+} is present.

C.T. for Mg^{2+}

1	**HYPOIODIDE TEST :** O.S. + KI + NaOH + I_2 solution till solution becomes yellow	Reddish brown precipitates of [$Mg(IO)_2$]	∴ Mg^{2+} is confirmed.
2	O.S. + $(NH_4)_2C_2O_4$ (Ammonium oxalate)	White precipitates of (MgC_2O_4) soluble in CH_3COOH	∴ Mg^{2+} is confirmed.
3	O.S. + NH_4OH	White precipitates soluble in NH_4Cl and HCl	∴ Mg^{2+} is confirmed.

***Hypoiodide solution* :** *To NaOH solution add equal amount of KI and few drops of I_2 solution till yellow colour is obtained.*

If no precipitation obtained in all above groups (i.e. from group Ist to VthA) then Vth B group (K^+, NH_4^+) is consider to be present.

GROUP Vth B :

Separation of K^+ and NH_4^+

1	O.S. + Freshly prepared Sodium cobaltinitrite	Yellow precipitates	∴ K^+ or NH_4^+ is present.
2	O.S. + NaOH (heat)	Evolution of NH_3 gas recognized by smell, alkaline action on litmus, turmeric paper turns to brown and gives a white fumes with a drop of conc. HCl	∴ NH_4^+ is present.
		No evolution of NH_3 gas	∴ K^+ is present.
3	O.S. + Nessler's reagent	Reddish brown precipitates	∴ NH_4^+ is present.

Sodium cobaltinitrite { $Na_3[Co(NO_2)_6]$ } : Take small quantity of solid sodium nitrite ($NaNO_2$), add few drops of cobaltnitrate[$Co(NO_3)_2$] solution and shake it. Add dilute acetic acid solution.

Now check this sodium cobaltinitrite solution with NH_4Cl solution : Take one drop of NH_4Cl solution add sodium cobaltinitrite solution in excess. It gives yellow precipitates, *means sodium cobiltinitrite solution is true. If no precipitation means that prepared sodium cobaltinitrate solution is not true.*

Nessler's reagent { $K_2[HgI_4]$ }: Take one drops of $HgCl_2$ solution, add to it KI solution drop wise with shaking till precipitates *just dissolve. Add an equal volume of NaOH and shake it.*
Now check with NH_4Cl solution. (one drop of NH_4Cl + nessler's reagent (excess) give yellowish brown precipitates, *If no precipitation means, prepared nessler's reagent is not true.)*

N.B. If the substance is soluble in water and phosphate or carbonate is found to be present. Then phosphate or carbonate of K^+ and NH_4^+ may be present. All other phosphate and carbonate are insoluble in water.

C. T. for K^+

1	O.S. + Freshly prepared Sodium cobaltinitrite	Yellow precipitates	∴ K^+ is confirmed.
2	O.S. + Picric acid (shake well)	Yellow precipitates	∴ K^+ is confirmed.
3	O.S. + Tartaric acid (shake well)	White precipitates	∴ K^+ is confirmed.
4	O.S. + Nessler's reagent	No precipitates	∴ K^+ is confirmed.

C. T. for NH_4^+

1	O.S. + Nessler's reagent	Reddish-brown precipitates	∴ NH_4^+ is confirmed.
2	O.S. + NaOH (heat)	Evolution of NH_3 gas recognized by smell, alkaline action on litmus, turmeric paper turns to brown and gives a white fumes with a drop of conc. HCl	∴ NH_4^+ is confirmed.

[H] INSOLUBLE PHOSPHATE SCHEME :

If given substance is insoluble in water and 1st and 2nd group are found to be absent and PO_4^{-3} is present then follow the **INSOLUBLE PHOSPHATE SCHEME** as given below.

Identification of positive radicals of group III[rd] A, III[rd] B, IV[th] and V[th] group

1	O.S. + NH_4Cl (solid) + 50% NH_4OH + H_2S solution in excess	Black precipitates	∴ Fe^{2+}, Fe^{3+}, Ni^{2+} & Co^{2+} is present.
		White precipitates	∴ Al^{3+}, Zn^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+} & Mg^{2+} is present.
		Green precipitates (O.S. is bluish green colour)	∴ Cr^{3+} is present.
		Buff coloured precipitates soluble in acetic acid	∴ Mn^{2+} is present.

Separation of black precipitation of Fe^{2+}, Fe^{3+}, Ni^{2+}, Co^{2+}

1	O.S. + NaOH sol^{n}. till just alkaline (excess)	Dirty green precipitates changing to reddish brown on exposure of air and insoluble in excess of NaOH	∴ Fe^{2+} is present.
		Reddish brown precipitates	∴ Fe^{3+} is present.
		Light green precipitates (O.S. is green)	∴ Ni^{2+} is present.
		Bluish precipitates (O.S. is red)	∴ Co^{2+} is present.

Separation of white precipitation of Al^{3+}, Zn^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+} & Mg^{2+}

1	O.S. + 2 – 3 drops of NaOH solution	(1) White precipitates soluble in excess of NaOH	∴ Al^{3+} or Zn^{2+} is present.
		(a) Reprecipitated on addition of NH_4OH solution	∴ Al^{3+} is present.
		(b) No reprecipitation on addition of NH_4OH solution	∴ Zn^{2+} is present.
		(2) White precipitates insoluble in excess of NaOH	∴ Ca^{2+}, Ba^{2+} Mg^{2+} & Sr^{2+} is present.

Identification of Ca^{2+}, Ba^{2+} Mg^{2+} & Sr^{2+}

1	O.S. + $CaSO_4$ solution	White precipitates immediately	∴ Ba^{2+} is present.
		White precipitates slowly or on warming	∴ Sr^{2+} is present.
		No precipitation even on warming	∴ Ca^{2+}, Mg^{2+} is present.
2	O.S. + Na_2CO_3 till precipitates formed + Acetic acid till just dissolves + ammonia oxalate (excess)	Reprecipitates	∴ Ca^{2+} is present.
		No reprecipitation	∴ Mg^{2+} is present.

[I] CONFIRMATIVE TEST FOR POSITIVE RADICAL :

Write confirmative test from earlier pages, whatever is applicable.

[J] WET TEST FOR NEGATIVE RADICALS (ANION) :

N.B: (a) When the substance is soluble in water, prepare its original solution in distilled water and detect the negative radical as under.

(b) When given substance is insoluble in water then negative radical can be detected only from **dry test.**

1	O.S. + $AgNO_3$ (shake well)	(A) White or yellowish white precipitates insoluble in dil. HNO_3 Take small quantity of above white precipitates of (A) + NH_4OH (shake well)	∴ Cl^-, Br^-, I^- is present.
		(i) White precipitates soluble in NH_4OH	∴ Cl^- is present.
		(ii) Yellowish precipitates soluble in excess of NH_4OH	∴ Br^- is present.
		(iii) Yellowish precipitates insoluble in excess of NH_4OH	∴ I^- is present.
		(B) Yellowish precipitates soluble in dilute HNO_3 without any effervences	∴ PO_4^{3-} is present.
		(C) Whitish grey precipitates soluble in dilute HNO_3 with effervences of CO_2 gas	∴ CO_3^{2-} is present.
		(D) Scarlet red precipitates soluble in dil. HNO_3	∴ CrO_4^{2-} or $Cr_2O_7^{2-}$ is present.
		(i) O.S. is yellow, turns orange on addition of dilute HCl	∴ CrO_4^{2-} is present.
		(ii) O.S. is orange, turns yellow on adding NaOH	∴ $Cr_2O_7^{2-}$ is present.
		(E) No precipitates	∴ SO_4^{2-} or NO_3^- is present.
2	O.S. + $BaCl_2$ solution	White precipitates insoluble in dilute HCl	∴ SO_4^{2-} is present.
		No precipitates	∴ NO_3^- is present.

N. B.: (a) Use $Ba(NO_3)_2$ instead of $BaCl_2$ if Ist group is present.

(b) Wet tests and C.T. for (–Ve) negative radical cannot be performed when the given substance is insoluble in water and it can be concluded only from the dry test.
[Insoluble CO_3^{2-}, O^{2-}, PO_4^{3-} and S^{-2} can be detected from dry test.]

1	Substance + dilute HCl	Effervences of CO_2 gas	∴ CO_3^{2-} is confirmed.
2	Substance + Conc. HCl (heat)	Evolution of a bad smelling gas (rotten eggs) turns to lead acetate paper black or silvery white	∴ S^{2-} is confirmed.
3	Substance + Conc. HNO_3 + Ammonium Molybdate (warm it)	Canary yellow precipitates	∴ PO_4^{3-} is confirmed.

4	Test for O^{-2} :	All the above negative radicals are found to be absent and the given substance is an amorphous and insoluble in water	∴ O^{2-} is confirmed.

[K] CONFIRMATORY TEST FOR NEGATIVE RADICALS :

C. T. for Cl^-

1	O.S. + Lead acetate	White precipitates soluble on heating and reprecipitated on cooling	∴ Cl^- is confirmed.
2	Substance+ MnO_2 (Powder) + Conc. H_2SO_4 and heat	Evolution of greenish yellow gas having pungent smell, bleaches the litmus paper (Blue/red litmus paper turns to white)	∴ Cl^- is confirmed.

C. T. for Br^-

1	O.S. + Lead acetate	White precipitates soluble on heating and reprecipitated. on cooling	∴ Br^- is confirmed.
2	O.S. + MnO_2 + Con. H_2SO_4 (Heat)	Reddish brown gas turns to wet starch paper yellow (orange)	∴ Br^- is confirmed.
3	O.S. + $CHCl_3$ + Cl_2 water (Shake well)	Yellow globules at bottom of test tube	∴ Br^- is confirmed.

C. T. for I^-

1	O.S. + Lead acetate	Yellow precipitates soluble on heating and becomes golden yellow	∴ I^- is confirmed.
2	O.S. + MnO_2 + Con. H_2SO_4 (heat)	Violet gas turns to wet starch paper blue	∴ I^- is confirmed.
3	O.S. + $CHCl_3$ + Cl_2 water (Shake well)	Violet globules at the bottom of the test tube	∴ I^- is confirmed.
4	O.S. + $HgCl_2$	Deep red precipitates soluble in excess of $HgCl_2$	∴ I^- is confirmed.

C. T. for PO_4^{-3}

1	O.S. + Conc. HNO_3 + $NH_4)_2MoO_4$ (Ammonium molybdate) in excess (warm it)	Canary yellow precipitates	∴ PO_4^{3-} is confirmed.
2	O.S. + Lead acetate	White precipitates	∴ PO_4^{3-} is confirmed.
3	O.S. + Magnesia mixture. ($MgCl_2$ + NH_4OH + NH_4Cl)	White precipitates	∴ PO_4^{3-} is confirmed.

C. T. for CO_3^{-2}

1	O.S. + $BaCl_2$ or $CaCl_2$	White precipitates soluble in acids with effervences of CO_2 gas	∴ CO_3^{2-} is confirmed.
2	O.S. + Lead acetate	White precipitates	∴ CO_3^{2-} is confirmed.
3	O.S. + dilute HCl (Shake well)	Effervences of CO_2 gas turns to lime water milky	∴ CO_3^{2-} is confirmed.

C. T. for NO_3^-

1	O.S. + Cu foil + Conc. H_2SO_4 heat slowly	Reddish brown fumes of NO_2 gas and no effect on wet starch paper	∴ NO_3^- is confirmed.
2	O.S. + Zn dust + NaOH (boil)	Evolution of NH_3 gas turns to wet red litmus paper blue	∴ NO_3^- is confirmed.
3	**Ring test :** O.S. + Conc. H_2SO_4 cool and add Freshly prepared $FeSO_4$ solution from the side of the test tube.	Brown ring at the junction of two liquids	∴ NO_3^- is confirmed.

C. T. for SO_4^{-2}

1	O.S. + $BaCl_2$	White precipitates insoluble in dilute acid	∴ SO_4^{2-} is confirmed.
2	O.S. + Lead acetate	White precipitates soluble in ammonium acetate solution	∴ SO_4^{2-} is confirmed.
3	O.S. + $Pb(NO_3)_2$	White precipitates insoluble in acid and soluble in hot ammonium acetate (NH_4Ac)	∴ SO_4^{2-} is confirmed.

C. T. for CrO_4^{-2}

1	Colour of original solution	Yellow	∴ CrO_4^{2-} is confirmed.
2	O.S. + $BaCl_2$ solution	Yellow precipitates soluble in dilute HNO_3	∴ CrO_4^{2-} is confirmed.
3	O.S. + Dilute HCl	Orange colour	∴ CrO_4^{2-} is confirmed.
4	O.S. + Lead acetate $[Pb(Ac)_2]$	Yellow precipitates	∴ CrO_4^{2-} is confirmed.

C. T. for $Cr_2O_7^{-2}$

1	Colour of Original solution	Orange	∴ $Cr_2O_7^{2-}$ is confirmed.
2	O.S. + $BaCl_2$	Yellow precipitates soluble in dilute HNO_3	∴ $Cr_2O_7^{2-}$ is confirmed.
3	O.S. + Lead acetate [$Pb(Ac)_2$]	Yellow precipitates	∴ $Cr_2O_7^{2-}$ is confirmed.

[L] EQUATION FOR CATION :

[L] EQUATION FOR ANION :

[M] FINAL CONCLUSION :

The given unknown substance contains :

Positive radical :

Negative radical :

Formula of the substance :

Name of the substance :

PREPARATION OF THE REAGENTS

Sodium cobaltinitrate reagent $Na_3[Co(No_2)_6]$	Take equal volume of $Co(NO_3)$ and $NaNO_2$ solution. Add dilute acetic acid to it. [Test with KI solution. It will give yellow precipitates]
Sodium Hypo iodide reagent [NaOI]	To NaOH solution add an equal volume of KI solution and then add few drops of I_2 solution till permanent yellow colour is obtained.
Nessler's reagent $K_2[HgI_4]$	Take few drops of $HgCl_2$ and add KI solution to it till precipitates just dissolved. And then add an equal volume of NaOH solution.
Magnesia mixture	Take 5 gm of $MgCl_2$ and dissolve in water (4-5 ml) to this solution add NH_4OH till it become alkaline. Then add NH_4Cl solution till the precipitates just dissolve.
Cobalt acetate $[Co(CH_3Coo)_2]$	Take cobaltinitrate solution $[Co(NO_3)_2]$ and add acetic acid to it.
Neutral $FeCl_3$	Take $FeCl_3$ solution and add NH_4OH to it, till reddish brown precipitates just form to start and then add water.

SOME CATION AND ANION CAN BE DIRECTLY CONFIRMED BY USING FOLLOWING TEST

Ni^{2+}: Substance + 50 % NH_4OH excess + DMG shake well, it gives scarlet red precipitates (Generally given substance is green or bluish green)

Cu^{2+}: Substance + 50 % NH_4OH shake well, it gives deep blue colouration. (Generally given substance is blue)

Fe^{2+}: Substance + Conc. NHO_3 + KCNS solution shake well, it gives blood red colour.

Mg^{2+}: Substance + Hypoiodide solution shake well, gives reddish brown precipitation.

PO_4^{-3}: Substance + Ammonium molybledum + conc.HNO_3 give cannery yellow precipitates. [If substance is blue (copper phosphate) then yellow precipitation is not clear yellow.]

I^- : Substance + conc. H_2SO_4 heat gently gives bluish violet gas.

NO_3^-: Substance + KI (solid) + starch solution + Zn dust gives blue colour.

S^{-2} : Substance (Insoluble in water) + Conc. HCl, heat, evolution of a bad smelling gas (rotten eggs) turns to lead acetate paper black or silvery white.

SO_4^{-2}: Substance + $BaCl_2$ gives white precipitates..