# INORGANIC QUALITATIVE ANALYSIS

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 known as a radical.

1. Positive radical or cation.
2. Negative radical or anion.

Usually the substance under analysis is subjected to:

**Dry Tests:** (For substance in the solid state only)

**Wet Tests:** (For the solutions prepared from solid state).

**DRY TESTS:**

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

**[A] PHYSICAL PROPERTIES:**

1. State
2. Colour
3. Odour
4. Solubility in water
5. Action of litmus paper

**[B] DRY TESTS FOR POSITIVE RADICAL OR CATION:**

1. Heating in a dry test tube
2. Mirror test
3. Charcoal cavity test
4. Cobalt nitrate test
5. Borax bead test
6. Flame test
7. Test of $NH_4^+$ (Sodium hydroxide test)

**[C] DRY TESTS FOR NEGATIVE RADICAL OR ANION:**

1. Test for $CO_3^{2-}$ and $S^{2-}$
2. Test for $Cl^-$, $Br^-$, $I^-$

3. Test for $NO_3^-$ (perform this test when halides are absent).
4. Test for phosphate ($PO_4^{3-}$)
5. Test for $CrO_4^{2-}$ and $Cr_2O_7^{2-}$
6. Test for $SO_4^{2-}$

## [A] PHYSICAL PROPERTIES:

| | | | |
|---|---|---|---|
| **1** | State | (a) Crystalline (Sandy powder)<br>(b) Amorphous (Soft powder) | Radicals like $K^+$, $NH_4^+$, $Cl^-$, $Br^-$, $NO_4^-$, $SO_4^{2-}$ may be present.<br>Radicals like $CO_3^{2-}$, $S^{2-}$, $PO_4^{3-}$, $O^{2-}$ may be present. |
| **2** | Colour | (a) White<br>(b) Black<br>(c) Green<br>(d) Blue<br>(e) Orange<br>(f) Light Pink<br>(g) Reddish Brown | Generally compounds of $Al^{3+}$, $Zn^{2+}$, $Ca^{2+}$, $Sr^{2+}$, $Ba^{2+}$, $Mg^{2+}$, $K^+$, $NH_4^+$ may be present.<br>Suplhides like CuS etc. may be present.<br>$Fe^{2+}$(ous) $Ni^{2+}$, $FeSO_4$ may be present.<br>$Cu^{2+}$, $Cu_3(PO_4)_2$ may be present.<br>$Cr_2O_7^{2-}$ may be present.<br>$MnCl_2$, or $MnSO_4$ may be present.<br>CdS may be present. |
| **3** | Odour (smell) | (a) Ammonical<br>(b) Smell of Rotten eggs (like $H_2S$) | Salts of $NH_4^+$ may be present.<br>Sulphides may be present. |
| **4** | Solubility in water | (a) Soluble<br>(b) Insoluble | $K^+$, $NH_4^+$, $Cl^-$, $Br^-$, $NO_3^-$, $SO_4^{-2}$ may be present.<br>$CO_3^{2-}$, $S^{2-}$, $PO_4^{3-}$, $O^{2-}$ may be present. |

| | | | |
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| **5** | Action on litmus paper (this test can be carried out only for water soluble compound) | (a) Acidic<br>(b) Basic<br>(c) Neutral | Salts of strong acid and weak base may be present.<br>Salts of weak acid and strong base may be present.<br>Salts of strong acid and strong base may be present. |

## [B] DRY TEST FOR POSITIVE RADICAL OR CATION:

| | | | |
|---|---|---|---|
| **1** | **Heating in a dry test tube** | | |
| | | (a) Substance melts | Salts of $K^+$, some salts containing water of crystallization may be present. |
| | | (b) Substance produces cracking noise. | Nitrates like $Pb(NO_3)_2$ crystalline salts like KBr may be present. |
| | | (c) Water condensing on the cooler part of the test tube. | Crystalline salts may be present. |
| | | (d) Sublimation takes place on the cooler part of the test tube (PERFORM MIRROR TEST) | $NH_4^+$ may be present. |
| | | (e) **CHANGE OF COLOUR** | |
| | | • Blackening at high temperature<br>• Yellow when hot and white when cold. | $Cu^{2+}$, $Mn^{2+}$, $Ni^{2+}$ may be present.<br>Salt of $Zn^{2+}$ may be present. |

| | | | |
|---|---|---|---|
| | | • Yellow | $Pb^{2+}$ may be present. |
| | | • Brown | Salt of $Cd^{2+}$ may be present. |
| | | • Green | $Cr_2O_7^{2-}$ may be present. |
| | | • No change<br>i.e. white remains white. | $Al^{3+}$, $Ca^{3+}$, $Ba^{2+}$, $Sr^{2+}$, $Mg^{2+}$ may be present. |
| | | (f) **EVOLUTION OF GAS** | |
| | | • Reddish brown gas turns starch paper yellow | $Br^-$ may be present. |
| | | • Reddish brown gas no effect on starch paper | $NO_3^-$ may be present. |
| | | • Greenish yellow gas, having pungent odour, bleaches litmus paper (blue to white) | $Cl^-$ may be present. |
| | | • Ammonia gas recognized by smell, alkaline to litmus, white fumes with a drop of conc. HCL and Turns turmeric paper brown. | $NH_4^+$ may be present. |
| | | • $SO_2$ gas (acidic pungent gas turns dichromate paper green) | $S^{2-}$ may be present. |
| | | • White fumes of $SO_3$ acidic to litmus and having pungent smell. | $SO_4^{2-}$ may be present. |
| | | • Substance is orange and gives sparks on heating and becomes dark green on cooling | $Cr_2O_7^{2-}$ may be present. |

| | | | |
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| **2** | **MIRROR TEST:** (Perform when white sublimation takes place) | | |
| | Sub. + $Na_2CO_3$ + Charcoal dust (Mixed thoroughly and heat the mixture in dry test tube) | Ammonia recognized by smell, fumes with a drop of conc. HCL turns turmeric paper brown. | $NH_4^+$ may be present. |
| **3** | **CHARCOAL CAVITY TEST:** | | |
| | Mix the powder with equal amount of sodium carbonate. Make a cavity in charcoal and moisten it with a drop of water. Put the mix in cavity along with a very small amount of charcoal dust and blow in reducing flame. | a) Deflagration (burning of charcoal) | $NO_3^-$, $Cr_2O_7^{2-}$ may be present. |
| | | b) Substance fuses and sinks in charcoal. | $K^+$ may be present. |
| | | c) Sublimation | $NH_4^+$ may be present. |
| | | d) White infusible mass which glows when heated (Perform $Co(NO_3)_2$ Test) | $Ca^{2+}$, $Sr^{2+}$, $Ba^{2+}$, $Al^{3+}$, $Zn^{2+}$, $Mg^{-2+}$ may be present. |
| | | e) Coloured infusible mass (Perfrom Borax Bead Test) | $Cu^{2+}$, $Mn^{2+}$, $Fe^{2+}$, $Ni^{2+}$, $Cr^{2+}$, $Cr_2O_7^{2-}$ may be present. |
| | | (f) **METALLIC BEADS** | |
| | | • White soft bead marking on paper and yellow encrustation | $Pb^{2+}$ may be present. |
| | | • Red scales | $Cu^{2+}$ may be present. |
| | | • Brown encrustation | $Cd^{2+}$ may be present. |
| | | • No bead, yellow when hot and when white when cold. | $Zn^{2+}$ may be present. |

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| **4** | **COBALT NITRATE TEST:** (This test should be performed only when white infusible mass is obtained in Charcoal cavity test) Put one to two drops of $Co(NO_3)_2$ solution on white infusible mass obtained in the charcoal cavity test and heat it strongly in oxidizing flame. | | |
| | | (a) Green mass | $Zn^{+2}$ may be present. |
| | | (b) Blues mass | $Al^{+3}$ or $PO_4^{-3}$ may be present. |
| | | (c) Pink mass | $Mg^{+2}$ may be present. |
| | | (d) No Green, blue or pink mass | $Ca^{+2}$, $Ba^{+2}$, $Sr^{+2}$ may be present. |
| **5** | **BORAX BEAD TEST:** (Perform this test only when the substance is coloured and leaves coloured infusible mass in the charcoal cavity test) (i.e. when any one of $Cu^{2+}$, $Fe^{2+}$, $Ni^{2+}$, $Mn^{2+}$, $Cr_2O_7^{2-}$, is suspected to be present). | | |
| | **Colour of bead from oxidizing flame** | **Colour of bead from reducing flame** | **Radicals** |
| (a) | Green | Green | $Cr_2O_7^{2-}$ may be present. |
| (b) | Green when hot and blue when cold. | Red and opaque | $Cu^{2+}$ may be present. |
| (c) | Yellow | Light green | $Fe^{2+}$ may be present. |
| (d) | Violet | Colourless | $Mn^{2+}$ may be present. |
| (e) | Brown | Grey and opaque | $Ni^{2+}$ may be present. |

| | | | |
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| **6** | **FLAME TEST:** Prepare a paste of the substance in conc. HCl or Conc. Solution of substance in con. HCl and perform flame test. | | |
| | | **Colour of the flame** | |
| | | (a) Brick red | $Ca^{2+}$ may be present. |
| | | (b) Crimson | $Sr^{2+}$ may be present. |
| | | (c) Light green | $Ba^{2+}$ may be present. |
| | | (d) Violet (Pink) | $K^{+}$ may be present. |
| | | (e) Green | $Cu^{2+}$ may be present. |
| | | (f) Bluish white | $Pb^{2+}$ may be present. |
| **7** | **TEST FOR $NH_4^+$:** (SODIUM HYDROXIDE TEST) Sub. + NaOH (heat) | | |
| | | Evolution of NH3 gas recognized by smell; alkaline to litmus, gives white fumes with drop of con. HCl. | $NH_4^+$ may be present. |

## [C] DRY TEST FOR NEGATIVE RADICAL (ANION)

| | | | |
|---|---|---|---|
| **1** | **TEST FOR $CO_3^{2-}$, $S^{2-}$:** | | |
| | Sub. + Dil HCl<br>Sub. + Dil HCl (heat) | Efferences of $CO_2$ gas | $CO_3^{2-}$ may be present.<br>$S^{2-}$ may be present. |
| **2** | **TEST FOR $Cl^-$, $Br^-$:** | | |
| | Sub. Con. $H_2SO_4$ + $MnO_2$ (heat) | • Greenish yellow gas with pungent smell and bleaches litmus paper.<br>• Reddish brown gas turning starch paper yellow. | $Cl^-$ may be present.<br>$Br^-$ may be present. |
| **3** | **TEST FOR $NO_3^-$:** (perform when halides are absent) | | |
| | Sub. + conc. $H_2SO_4$ + Cu foil (heat) | • Reddish brown gas, no effect on starch paper. | $NO_3^-$ may be present. |

| | | | |
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| **4** | **TEST FOR $PO_4^{3-}$:** | | |
| | Dissolve Sub in few drops con. $HNO_3$, and excess $(NH_4)_2MoO_4$ (Ammonium molybdate) solution. (heat) | Canary yellow ppt | $PO_4^{-3}$ may be present. |
| **5** | **TEST FOR $Cr_2O_7^{2-}$:** | | |
| | If given sub is orange, add dil. NaOH solution to its aqueous solution | Solution turns yellow | $Cr_2O_7^{2-}$ may be present. |
| **6** | **TEST FOR $SO_4^{2-}$:** | | |
| | Crystalline sub. + $H_2O$ | Soluble | $SO_4^{2-}$ may be present. |
| **7** | If all the above radicals are absent and the given salt is an amorphous solid insoluble in the water then $O^{2-}$ may be present | | |

**Note:** Sulfates of Lead, Calcium, Barium and Strontium are sparingly soluble in water.

## [D] CONCLUSION FROM DRY TEST:

Probable Cation:

Probable Anion:

## [E] PREPARATION OF SOLUTION FOR WET TEST:

| | | | |
|---|---|---|---|
| (a) | Sub. + distlled $H_2O$ (warm if it remains insoluble) | Soluble | O.S. (Possibility of $K^+$, $NH_4^+$, $NO3^-$, $SO_4^{2-}$, $Cl^-$, $Br^-$) |
| (b) | Sub. + dil HCl (Heat if it is insoluble at room temp.) | Soluble | O.S. (Possibility of $CO_3^{2-}$, $PO_4^{3-}$) |
| (c) | Sub. + conc. HCl try to dissolve as much of substance as possible decant the solution and dilute with distilled water. | Soluble | O.S. |

**Note:**

- If insoluble sulphide is present boil the salt with HCl to expel $H_2S$ gas till the lead acetate paper does not turn black.
- If the salt is insoluble in water and $PO_4^{3-}$ is detected then follow the 'Phosphate scheme' for the identification of the metallic radical after the 1st and 2nd groups.
- 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 RADICAL (CATION):

| | | | |
|---|---|---|---|
| **1** | O.S. + Dil HCl | • White ppts. | Gr. I present i.e. $Pb^+$ present |
| **2** | O.S. dil. HCl<br>$H_2S$ (excess) | • Coloured ppts or white turbidity.<br>• Black or brown ppt.<br>• Yellow ppt. | Gr. II is present.<br>$Cu^{2+}$, $Pb^{2+}$ may be present.<br>$Cd^{+2}$ may be present. |

**Note:** If $PO_4^{3-}$ is present & substance is insoluble in $H_2O$ and 1st and 2nd Groups are absent then follow the insoluble phosphate scheme (**page no. 19**)

| | | | |
|---|---|---|---|
| **3** | O.S. + $NH_4Cl$ (excess or in solid form) + $NH_4OH$ till the solution becomes alkaline | White or coloured ppt.<br>• White gelatinous ppt.<br>• Green ppt. | Gr. IIIA is present<br>$Al^{3+}$ present<br>$Fe^{+2}$ present |
| **4** | O.S. + $NH_4Cl$ + $NH_4OH$ + $H_2S$ | White, buff or black ppt.<br>(a) White ppts, insoluble in $CH_3COOH$<br>(b) Buff ppt soluble in $CH_3COOH$<br>(c) Black ppts | Gr. IIIB present i.e. $Mn^{2+}$, $Zn^{2+}$, $Ni^{2+}$ present<br>$Zn^{2+}$ present<br>$Mn^{2+}$ present<br>$Ni^{2+}$ present |

| | | | |
|---|---|---|---|
| **5** | O.S. + $NH_4Cl$ + $NH_4OH$ + $(NH_4)_2CO_3$ | White ppts soluble in HCl with effervescence | Gr. IV present i.e. $Ca^{2+}$, $Ba^{2+}$, $Sr^{2+}$ present. |
| **6** | O.S. + $NH_4Cl$ + $NH_4OH$ + $Na_2HPO_4$ | White ppts | Gr. V is present i.e. $Mg^{2+}$ present |
| If all the above groups (i.e. from gr. I to V) are absent then $K^+$ or $NH_4^+$ may be present. | | | |

**[G] SEPERATION OF GROUP RADICALS, GENERAL SCHEME:**

## GROUP – I

| | | |
|---|---|---|
| O.S. + dil HCl | White ppt. | Gr. I present i.e. $Pb^{2+}$ present |
| Above white ppts + $NH_4OH$ | ppts unaffected | $Pb^{2+}$ present |

**C.T. For $Pb^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $K_2CrO_4$ | Yellow ppts ($PbCrO_4$) soluble in hot NaOH | $Pb^{2+}$ is confirmed. |
| (2) | O.S. + Kl | Yellow ppts ($PbI_2$) soluble in hot water, recrystallises into golden yellow needle on cooling. | $Pb^{2+}$ is confirmed. |
| (3) | O.S. + NaOH | White ppts of $[Pb(OH)_2]$ soluble in excess NaOH | $Pb^{2+}$ is confirmed. |

## GROUP – II

Identification of $Cu^{2+}$, $Pb^{2+}$

| | | |
|---|---|---|
| O.S. + NaOH | • Bluish ppts turns black on heating<br>• White ppts of $[Pb(OH)_2]$ soluble in excess NaOH | $Cu^{2+}$ is present<br>$Pb^{2+}$ is confirmed. |

Identification of group II, $Cd^{2+}$ (Yellow ppt. From grp.-II divided into two parts)

| | | |
|---|---|---|
| Yellow ppts + Conc. HCl | ppts soluble | $Cd^{2+}$ is present |
| Yellow ppts + $(NH_4)_2S_x$ (ammonium poly sulphide) | ppts insoluble | $Cd^{2+}$ is present |

**C.T. For $Cu^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + NaOH | Blue ppts turns black on heating | $Cu^{2+}$ is confirmed. |
| (2) | O.S. + $NH_4OH$ | Bluish ppts soluble in excess $NH_4OH$ giving deep blue solution | $Cu^{2+}$ is confirmed. |
| (3) | O.S. + KI | Dirty green ppts ($Cu_2I_2$) appearing brown due to liberation of $I_2$ | $Cu^{2+}$ is confirmed. |
| (4) | O.S. + $K_4[Fe(CN)_6]$ | Reddish brown ppts $Cu_2[Fe(CN)_6]$ | $Cu^{2+}$ is confirmed. |

**C.T. For $Cd^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $Na_2CO_3$ | White ppts of $CdCO_3$ | $Cd^{2+}$ is confirmed. |
| (2) | O.S. + NaOH | White ppts Insoluble in excess NaOH | $Cd^{2+}$ is confirmed. |
| (3) | O.S. + $H_2S$ | Yellow ppts Insoluble in yellow ammonium poly sulphide | $Cd^{2+}$ is confirmed. |

## GROUP – IIIA

| | | |
|---|---|---|
| O.S. + $NH_4Cl$ (excess) + $NH_4OH$ till alkaline | (a) Gelatinous white ppts (O.S. Colourless) | $Al^{3+}$ is present. |
| | (b) Dirty green ppts turns brown on surface due to exposure in air | $Fe^{2+}$ (ous) present |

**C.T. For $Al^{3+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + NaOH | White gelatinous ppts soluble in excess and reprecipitated by $NH_4Cl$ | $Al^{3+}$ is confirmed. |
| (2) | O.S. + $NH_4OH$ | White ppts of $Al(OH)_3$ | $Al^{3+}$ is confirmed. |
| (3) | O.S. + $K_4[Fe(CN)_6]$ | No ppts | $Al^{+3}$ is confirmed. |

**C.T. For $Fe^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $K_3[Fe(CN)_6]$ | Dark blue ppts | $Fe^{2+}$ is confirmed. |
| (2) | O.S. + $K_4[Fe(CN)_6]$ | Bluish white ppts of $FeK_2[Fe(CN)_6]$ | $Fe^{2+}$ is confirmed. |
| (3) | O.S. + NaOH | Dirty green ppts of Fe(OH) turning reddish brown on expose in air. | $Fe^{2+}$ is confirmed. |

# GROUP – III B

| | | | |
|---|---|---|---|
| O.S. + $NH_4Cl$ + $NH_4OH$ + $H_2S$ | (a) | White ppts insoluble in acetic acid | $Zn^{2+}$ is present. |
| | (b) | Pink or white or Buff ppts soluble in acetic acid | $Mn^{2+}$ is present. |
| | (c) | Black ppts | $Ni^{2+}$ is present. |

**C.T. For $Zn^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $K_4[Fe(CN)_6]$ | White ppts of $Zn_2[Fe(CN)_6]$ insoluble in dil. HCl | $Zn^{2+}$ is confirmed. |
| (2) | O.S. + $NH_4OH$ or NaOH | White ppts of $Zn(OH)_2$ soluble in excess. | $Zn^{2+}$ is confirmed. |
| (3) | O.S. + $K_3[Fe(CN)_6]$ | Orange ppts soluble in dil. HCl | $Zn^{2+}$ is confirmed. |

**C.T. For $Mn^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $K_4[Fe(CN)_6]$ | No. ppts if O.S. is acidic | $Mn^{2+}$ is confirmed. |
| (2) | O.S. + KCN | Pink white ppts | $Mn^{2+}$ is confirmed. |
| (3) | O.S. + $PbO_2$ + Con. $HNO_3$ boil & dilute with water and allow it settle | Pink colour of $KMnO_4$ | $Mn^{2+}$ is confirmed. |

**C.T. For $Ni^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $Br_2$ water + NaOH in excess | Black ppts | $Ni^{2+}$ is confirmed. |
| (2) | O.S. + Dimethylglyoxime ($C_4H_7O_2N_2$) + $NH_4OH$ | Brick red ppts | $Ni^{2+}$ is confirmed. |
| (3) | O.S. + NaOH or $NH_4OH$ | Light green ppts soluble in excess. | $Ni^{2+}$ is confirmed. |

## GROUP – IV

| | | |
|---|---|---|
| O.S. + $NH_4Cl$ + $NH_4OH$ + $(NH_4)_2CO_3$ | White ppts soluble in HCl with effervescence | $Ca^{2+}$, $Ba^{2+}$, $Sr^{2+}$ present. |
| SEPERATION OF $Ca^{2+}$, $Ba^{2+}$ and $Sr^{2+}$ (GROUP IV): O.S. + $CaSO_4$ | | |
| | White ppts immediately<br>White ppts on warming<br>No ppts | $Ba^{2+}$ is present.<br>$Sr^{2+}$ is present.<br>$Ca^{2+}$ is present. |

**C.T. For $Ca^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $(NH_4)_2CO_3$ | White ppts soluble in acid with effervescence of $CO_2$ | $Ca^{2+}$ is confirmed. |
| (2) | O.S. + $(NH_4)_2C_2O_4$ | White ppts soluble in acid | $Ca^{2+}$ is confirmed. |

**C.T. For $Sr^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $K_2CrO_4$ | Yellow ppts soluble in acetic acid | $Sr^{2+}$ is confirmed. |
| (2) | O.S. + $(NH_4)_2C_2O_4$ | White ppts soluble in dil. acetic acid. | $Sr^{2+}$ is confirmed. |

**C.T. For $Ba^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $(NH_4)_2C_2O_4$ | White ppts soluble in dil. acetic acid | $Ba^{2+}$ is confirmed. |
| (2) | O.S. + dil. $K_2CrO_4$ | Yellow ppts turns orange by addition of acid. | $Ba^{2+}$ is confirmed. |

## GROUP – V

| | | | |
|---|---|---|---|
| (1) | O.S. + $NH_4Cl$ + $NH_4OH$ + $Na_2HPO_4$ | White ppts | $Mg^{2+}$ is confirmed. |

**C.T. For $Mg^{2+}$:**

| | | | |
|---|---|---|---|
| (1) | **HYPO-IODIDE TEST**: To NaOH adds equal amount of KI and few drops of $I_2$ solution till yellow colour is obtained. Few drops of above solution + O.S. | Reddish brown ppts | $Mg^{2+}$ is confirmed. |
| (2) | O.S. + NaOH | White ppts soluble in $NH_4Cl$ and HCl | $Mg^{2+}$ is confirmed. |
| (3) | O.S. + $NH_4OH$ | White ppts soluble in $NH_4Cl$ and HCl | $Mg^{2+}$ is confirmed. |

**Separation of $NH_4^+$ and $K^+$**

| | | | |
|---|---|---|---|
| (1) | O.S. + Sodiumcobaltinitrite (Freshly prepared) | Yellow ppts | $NH_4^+$ or $K^+$ may be present. |
| (2) | O.S. + NaOH (Heat) | • $NH_3$ gas is evolved turns litmus paper blue.<br>• No evolution of gas | $NH_4^+$ is present.<br>$K^+$ is present. |

**To prepapre sodium cobaltinitrite: -** Take solid $NaNO_2$ 1–2 gms. Add to it few drops of $Co(NO_3)_2$ solution and then add acetic acid till reddish brown colour solution is obtained. Test with KI solution give yellow ppt.

**Note:** Phosphates and carbonates of $NH_4^+$ or $K^+$ are soluble in water and all other phosphates and carbonates are insoluble in water.

**C.T. For $NH_4^+$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + Nessler's reagent ($HgCl_2$ + KI till ppt. dissolved + NaOH in equal amount) | Brown ppts | $NH_4^+$ is confirmed. |
| (2) | O.S. + Sodiumcobaltinitrite $[Na_3Co(NO_2)_6]$ (Freshly prepared) | Yellow ppts | $NH_4^+$ is confirmed. |

**C.T. For $K^+$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + Sodiumcobaltinitrite | Yellow ppts | $K^+$ is confirmed. |
| (2) | O.S. + Picric acid | Yellow ppts | $K^+$ is confirmed. |
| (3) | O.S. + Tartaric acid | Yellow ppts | $K^+$ is confirmed. |

**Note:**

- Use $Ba(NO_3)_2$ instead of $BaCl_2$ if 1st group is present.
- Wet-test for negative radical cannot be performed when the given substance is insoluble in water and C.T. for negative radical cannot be performed. It can be concluded only from the dry test.

## [I] WET TESTS FOR NEGATIVE RADICAL (ANION)

When the substance is soluble in water, prepare the original solution in distilled water and detect the negative radical.

| | | | |
|---|---|---|---|
| **O.S. + $AgNO_3$** | **(a)** | **White or yellowish white ppt. Insoluble in dit. $HNO_3$** | **$Cl^-$, $Br^-$ present.** |
| SEPARATION OF $Cl^-$, $Br^-$, $I^-$ | | | |
| ppts obtained + $NH_4OH$ | • White ppts Dissolved<br>• Yellowish ppts soluble in excess of $NH_4OH$ | | $Cl^-$ is present.<br>$Br^-$ is present. |
| | **(b)** | Yellowish ppts soluble in dil. $HNO_3$ | $PO_4^{3-}$ is present. |
| | **(c)** | Whitish grey ppts soluble in dil. $HNO_3$ | $CO_3^{2-}$ is present. |
| | **(d)** | Black or brown ppts soluble in dil. $HNO_3$ | $S^{2-}$ is present. |
| | **(e)** | Scarlet red ppts soluble in dil. $HNO_3$ | $Cr_2O_7^{2-}$ is present. |
| | | • O.S. is orange colour turns yellow on adding NaOH | $Cr_2O_7^{2-}$ is present. |
| | **(f)** | No ppts | $SO_4^{2-}$ or $NO_3^-$ is present. |
| SEPARATION OF $SO_4^{2-}$ & $NO_3^-$ | | | |
| O.S. + $BaCl_2$ | • White ppts insoluble in acid.<br>• No ppts | | $SO_4^{2-}$ is present.<br>$NO_3^-$ is present. |

**C.T. For $Cl^-$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + Lead acetate ($PbAc_2$) | White ppts soluble on heating and reprecipitated on cooling. | $Cl^-$ is confirmed. |
| (2) | O.S. + $MnO_2$ + conc. $H_2SO_4$ (Heat) | Greenish yellow gas having irritating smell bleaches the litmus paper | $Cl^-$ is confirmed. |
| (3) | O.S. + $CHCl_3$ + $Cl_2$ water (shake well) | Colourless layer | $Cl^-$ is confirmed. |

**C.T. For $Br^-$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + Lead acetate $Pb(CH_3COO)_2$ | White ppts soluble on heating | $Br^-$ is confirmed. |
| (2) | O.S. + $MnO_2$ + conc. $H_2SO_4$ (Heat) | Reddish brown gas turns starch paper yellow | $Br^-$ is confirmed. |
| (3) | O.S. + $CHCl_3$ + $Cl_2$ water (shake well) | Yellow globules at the bottom of test tube | $Br^-$ is confirmed. |

**C.T. For $PO_4^{3-}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + conc. $HNO_3$+ $(NH_4)MoO_4$ in excess (ammoniummolybdate) heat | Canary yellow ppts | $PO_4^{3-}$ is confirmed. |
| (2) | O.S. + Lead acetate | White ppts | $PO_4^{3-}$ is confirmed. |
| (3) | O.S. + Magnesia mixture ($MgCl_2$ + $NH_4OH$ + $NH_4Cl$) | White ppts | $PO_4^{3-}$ is confirmed. |

**C.T. For $CO_3^{2-}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $BaCl_2$ | White ppts soluble in acids with effervescence in $CO_2$ | $CO_3^{2-}$ is confirmed. |
| (2) | O.S. + Lead acetate | White ppts | $CO_3^{2-}$ is confirmed. |
| (3) | O.S. + $AgNO_3$ | Greyish white ppts soluble in $HNO_3$ with effervescence of $CO_2$ gas. | $CO_3^{2-}$ is confirmed. |
| (4) | O.S. + dli. Acid | Effervescence of $CO_2$ gas turns limewater milky. | $CO_3^{2-}$ is confirmed. |

**C.T. For $S^{2-}$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + $PbAC_2$ (Lead acetate) | White ppts soluble in dil. $HNO_3$ | $S^{2-}$ is confirmed. |
| (2) | O.S. + $HgCl_2$ | Black ppts | $S^{2-}$ is confirmed. |
| (3) | O.S. + Sodiumnitropruside | Violet colouration | $S^{2-}$ is confirmed. |

**C.T. For $NO_3^-$:**

| | | | |
|---|---|---|---|
| (1) | O.S. + Cu-Foil + conc. $H_2SO_4$ & Heat | Reddish brown gas, no effect on starch paper | $NO_3^-$ is confirmed. |
| (2) | **RING TEST:** O.S. + Conc. $H_2SO_4$ cool and slowly 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. + $PbAC_2$ (Lead acetate) | White ppts soluble in ammoniumacetate ($NH_4AC_2$) | $SO_4^{2-}$ is confirmed. |
| (2) | O.S. + $BaCl_2$ | White ppts insoluble in dil. acid. | $SO_4^{2-}$ is confirmed. |
| (3) | O.S. + $Pb(NO_3)_2$ | White ppts insoluble in acid and soluble in $NH_4AC_2$ (Ammonium acetate) | $SO_4^{2-}$ is confirmed. |

**C.T. For $Cr_2O_7^{2-}$:**

| | | | |
|---|---|---|---|
| (1) | Colour of solution | Orange | $Cr_2O_7^{2-}$ is confirmed. |
| (2) | O.S. + $PbAC_2$ | Yellow ppts | $Cr_2O_7^{2-}$ is confirmed. |
| (3) | O.S. + $BaCl_2$ | Yellow ppts | $Cr_2O_7^{2-}$ is confirmed. |

**[L] FINAL CONCLUSION:**

| CATION | ANION | FORMULA | NAME |
|---|---|---|---|
| | | | |

## INSOLUBLE PHOSPHATE SCHEME:

| | | |
|---|---|---|
| **O.S. + $NH_4Cl$ + $NH_4OH$ (till alkaline) + $H_2S$ water in exess** | **(a) Black ppts** | $Fe^{2+}$, $Fe^{3+}$, $Ni^{2+}$, is present |
| | **(b) White ppts** | $Al^{3+}$, $Zn^{2+}$, $Ca^{2+}$, $Mg^{2+}$, $Ba^{2+}$, $Ca^{2+}$, $Sr^{+}$ is present. |
| | **(c) Buff coloured ppts soluble in acetic acid** | $Mn^{2+}$ is present. |
| **Separation of Black ppt. ($Fe^{2+}$, $Ni^{2+}$):** | | |
| O.S. + NaOH | • Dirty green ppts changing to reddish brown when exposed to air | $Fe^{2+}$ is present. |
| | • Light green ppts (O.S. Green colour) | $Ni^{2+}$ is present. |
| **Separation of White ppt. ($Al^{3+}$, $Zn^{2+}$, $Ca^{2+}$, $Ba^{2+}$, $Sr^{2+}$, $Mg^{2+}$):** | | |
| O.S. NaOH<br><br>Above solution + $NH_4Cl$ | (1) White ppts soluble in excess NaOH | $Al^{3+}$, or $Zn^{2+}$ is present. |
| | • Reprecipitation | $Al^{3+}$ is present. |
| | • No ppts | $Zn^{2+}$ is present. |
| | (2) White ppts insoluble in excess NaOH | $Ca^{2+}$, $Ba^{2+}$, $Sr^{2+}$, $Mg^{2+}$, is present. |
| Identification of $Ca^{2+}$, $Ba^{2+}$, $Sr^{2+}$, $Mg^{2+}$: | | |
| O.S. + $CaSO_4$ | • White ppts immediately | $Ba^{2+}$ is present. |
| | • White ppts slowly or on warming | $Sr^{2+}$ is present. |
| | • No ppts even on warming | $Ca^{2+}$, $Mg^{2+}$ is present. |
| Identification of $Ca^{2+}$, $Mg^{2+}$: | | |
| O.S. + $Na_2CO_3$ till ppts formed + acetic acid till | • Reprecipitation | $Ca^{2+}$ is present. |
| | • No ppts | $Mg^{2+}$ is present. |

| | | |
|---|---|---|
| ppts just dissolve + Ammonium oxalate | | |
| Distinguish between $Ba^{2+}$ and $Sr^{2+}$ by following test: | | |
| O.S. + $Na_2CO_3$ till ppts obtained + Acetic acid (excess) + $K_2CrO_4$ | • Yellow ppts<br>• No ppts | $Ba^{2+}$ is present.<br>$Ca^{2+}$ is present. |