NEET | JEE ESSENTIALS

Class XI

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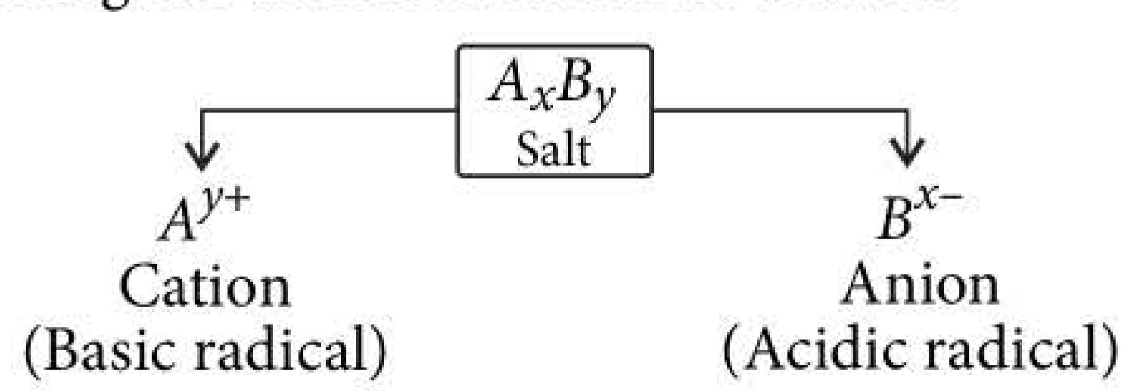
Unit 9 PRINCIPLES RELATED TO PRACTICAL CHEMISTRY

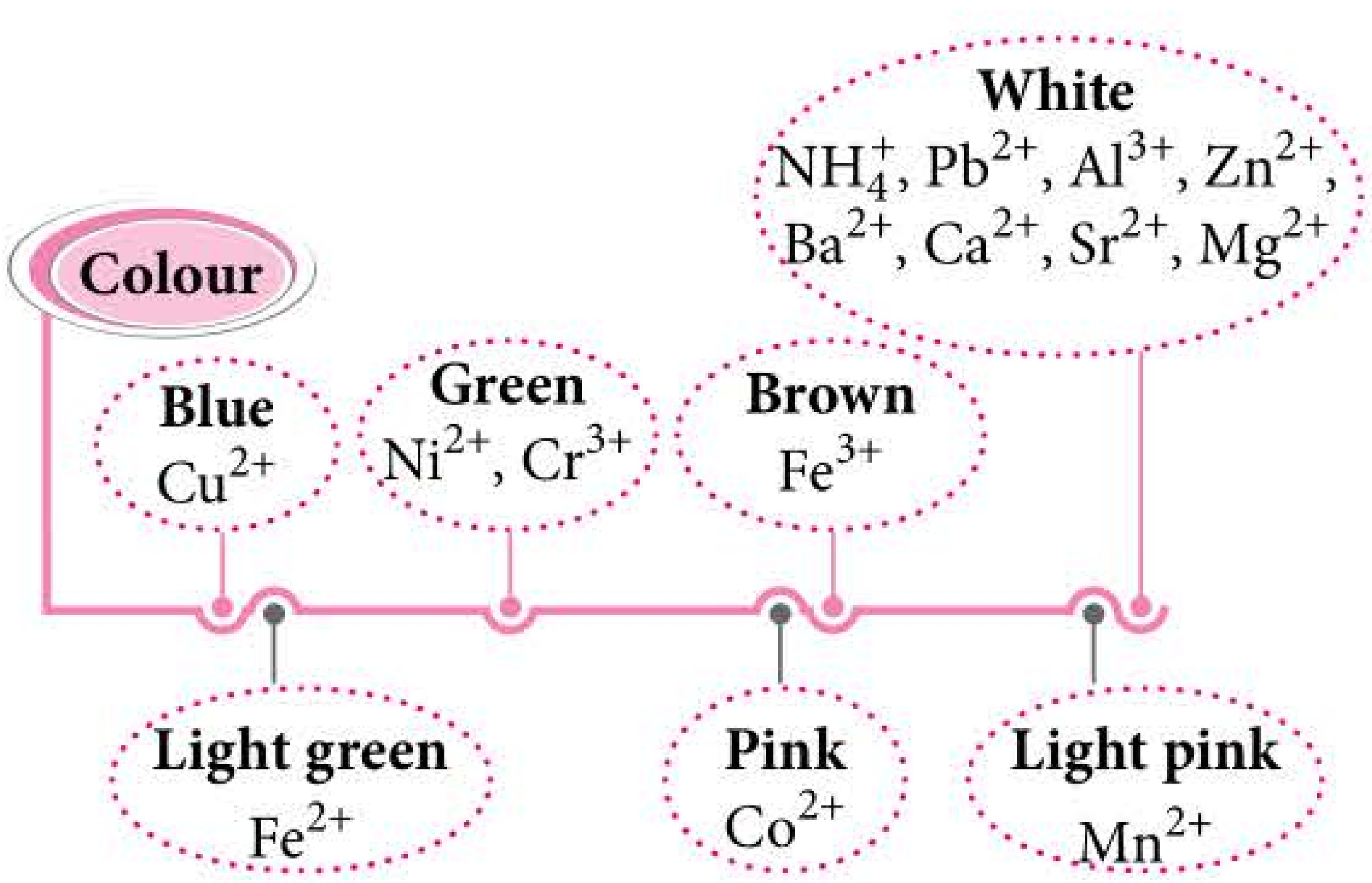
SALT ANALYSIS

The qualitative salt analysis deals with the identification of acidic radicals (anions) and basic radicals (cations) in an inorganic salt or in a mixture of salts.

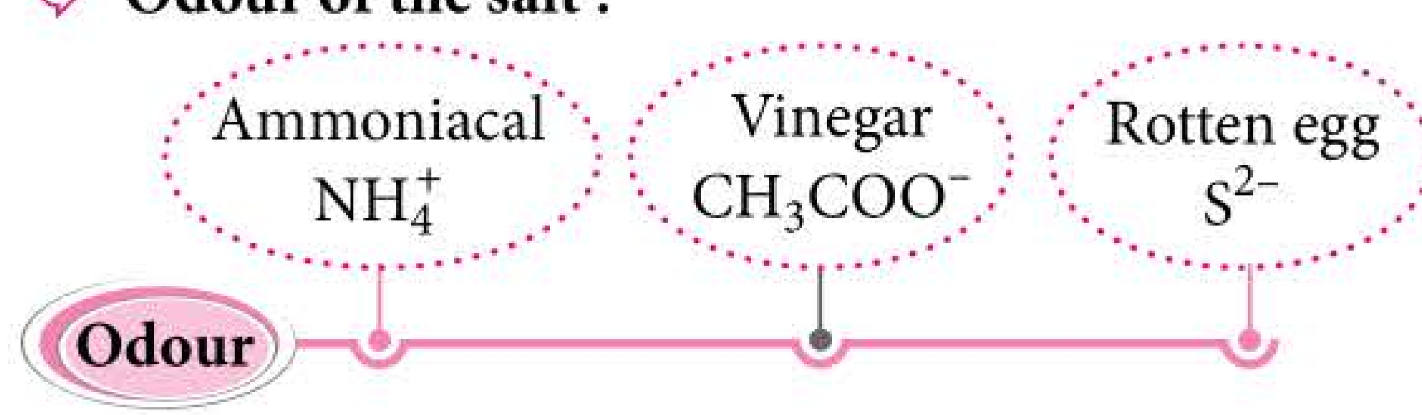


Preliminary Tests

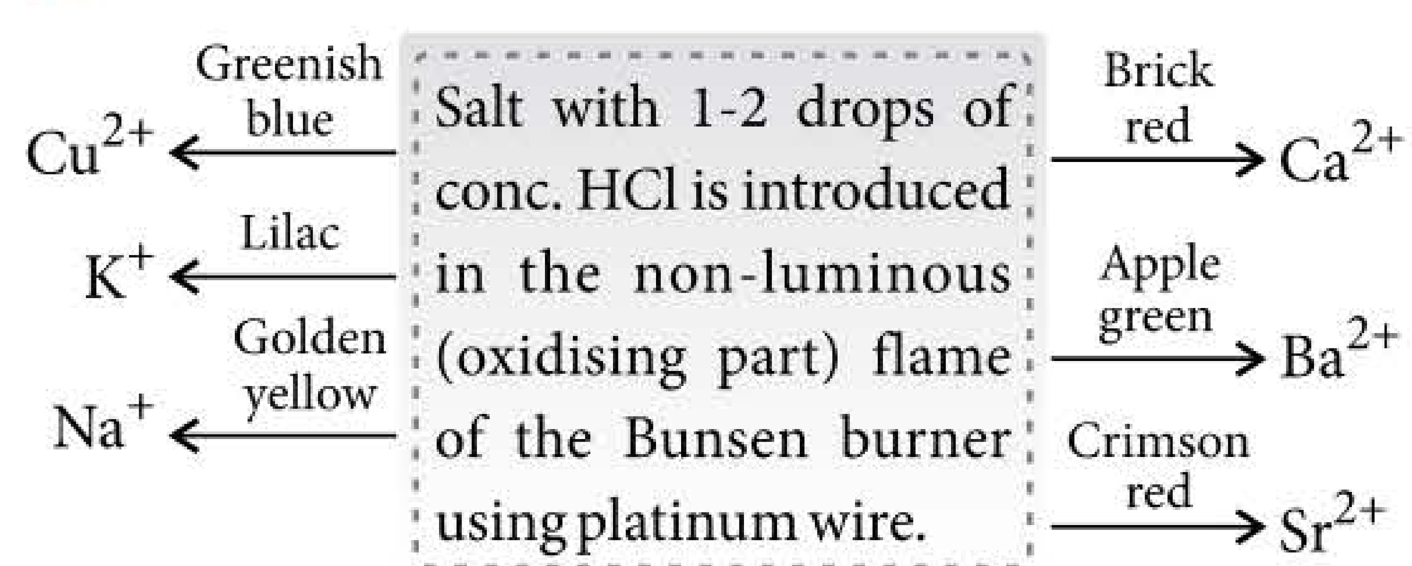
- Note the state (amorphous or crystalline) and colour of the salt.
- **Colour of the salt :**



- **Odour of the salt :**



- **Flame test :**



- **Borax bead test :**
  - Borax is heated on a loop of Pt wire, colourless glassy bead of sodium metaborate and boric anhydride is formed.

$$Na_2B_4O_7\cdot 10H_2O \xrightarrow{\Delta} Na_2B_4O_7 \xrightarrow{\Delta} \underbrace{2NaBO_2 + B_2O_3}_{\text{Glassy bead}}$$

  - Coloured salts are then heated on the glassy bead, coloured metaborate is formed in the oxidising flame.

Colour of bead in oxidising flame	Ion indicated
Green in hot, light brown in cold	Copper
Pinkish violet in both hot and cold	Manganese
Yellowish brown in hot and pale yellow in cold	Iron
Brown in hot and pale brown in cold	Nickel

ANIONS OR ACIDIC RADICALS

Anions

- **First group :** CO_3^{2-}, S^{2-}, SO_3^{2-}, CH_3COO^-, $S_2O_3^{2-}$, NO_2^-
- **Second group :** Br^-, Cl^-, I^-, NO_3^-, $C_2O_4^{2-}$
- **Third group :** SO_4^{2-}, PO_4^{3-}

First group :
Group reagent : dil. HCl

Salt + dil. HCl

Effervescence or evolution of gas shows presence of 1^{st} group

- Colourless gas with suffocating smell with yellow ppt. of sulphur $\xrightarrow{\text{Pass through } K_2Cr_2O_7}$ Blackish green solution $\longrightarrow$ $S_2O_3^{2-}$ confirmed
- Brown gas (NO_2) $\xrightarrow{\text{Starch iodide paper}}$ Blue $\longrightarrow$ NO_2^- confirmed
- Colourless gas with vinegar smell (CH_3COO^- may be present) Salt solution + $FeCl_3$ (neutral) $\longrightarrow$ $(CH_3COO)_3Fe$ (Blood red colour solution) $\longrightarrow$ CH_3COO^- confirmed
- Colourless gas with suffocating smell (SO_2) $\xrightarrow{\text{Heat and pass through } K_2Cr_2O_7}$ Solution turns green due to $Cr_2(SO_4)_3$ $\longrightarrow$ SO_3^{2-} confirmed
- Colourless gas with rotten egg smell (H_2S gas) $\xrightarrow{\text{Lead acetate}}$ PbS (Black ppt.) $\longrightarrow$ S^{2-} confirmed
- Colourless and odourless gas (CO_2 gas) $\xrightarrow{\text{Lime water}}$ $CaCO_3$ (milky) (CO_3^{2-} or HCO_3^- may be present)
- Confirmation test for HCO_3^- & CO_3^{2-}

  Salt + water → boil and pass through lime water
  - Lime water turns milky ↓ HCO_3^- confirmed
  - Lime water does not turn milky ↓ CO_3^{2-} confirmed

Second group :
Group reagent : Conc. H_2SO_4

Salt + conc. H_2SO_4

↓ Heat if required

Effervescence or evolution of gas shows presence of 2^{nd} group

- Colourless gas with pungent smell (HCl gas) → (Glass rod dipped in NH_4OH is exposed) → White fumes (NH_4Cl) → Cl^- may be present
- Brown gas → (Pieces of copper turning)
  - More brown fumes with blue solution → NO_3^- may be present
  - Not a blue solution → Br^- may be present
- Violet gas (I_2) → ($MnO_2 + H_2SO_4$) → Violet colour intensifies (more I_2) → I^- may be present
- Colourless and odourless gas (CO + CO_2) → (Burn) → Blue flame → $C_2O_4^{2-}$ may be present

Confirmatory tests of acid radicals :

Confirmatory tests for acid radicals of group II

Nitrate (NO_3^-)

Brown ring test : On treating aqueous solution of salt with freshly prepared solution of ferrous sulphate and concentrated sulphuric acid, gives a brown ring at the junction of two liquids.

$$\underset{\text{Salt}}{NaNO_3} + H_2SO_4 \longrightarrow NaHSO_4 + HNO_3$$

$$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + 4H_2O + 2NO$$

$$[Fe(H_2O)_6]SO_4 + NO \longrightarrow \underset{\text{Brown ring}}{[Fe(H_2O)_5NO]SO_4} + H_2O$$

Oxalate ($C_2O_4^{2-}$)

On acidifying sodium carbonate extract with acetic acid and on adding cadmium chloride solution gives white precipitate. Filter and dissolve the precipitate in dilute sulphuric acid and add few drops of potassium permanganate solution. The colour of potassium permanganate is discharged, indicates the presence of oxalate.

$$\underset{\text{Sodium carbonate extract}}{Na_2C_2O_4} + CaCl_2 \longrightarrow \underset{\text{White ppt.}}{CaC_2O_4\downarrow} + 2NaCl$$

$$CaC_2O_4 + H_2SO_4 \longrightarrow H_2C_2O_4 + CaSO_4$$

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$\begin{matrix} COOH \\ | \\ COOH \end{matrix} + [O] \longrightarrow 2CO_2 + H_2O$$

Chloride (Cl^-)

Chromyl chloride test : On heating salt with concentrated sulphuric acid in the presence of potassium dichromate, deep red vapours of chromyl chloride are evolved.

$$\underset{\text{Salt}}{NaCl} + H_2SO_4 \longrightarrow NaHSO_4 + HCl$$

$$K_2Cr_2O_7 + 2H_2SO_4 \longrightarrow 2KHSO_4 + 2CrO_3 + H_2O$$

$$CrO_3 + 2HCl \longrightarrow \underset{\substack{\text{Chromyl chloride}\\\text{(Red vapours)}}}{CrO_2Cl_2\uparrow} + H_2O$$

These vapours on passing through sodium hydroxide solution give yellow solution of sodium chromate.

$$CrO_2Cl_2 + 4NaOH \longrightarrow \underset{\text{Yellow colour}}{Na_2CrO_4} + 2NaCl + 2H_2O$$

The yellow solution on neutralising with acetic acid and on addition of lead acetate gives yellow precipitate of lead chromate.

$$Na_2CrO_4 + (CH_3COO)_2Pb \longrightarrow \underset{\text{Yellow ppt.}}{PbCrO_4\downarrow} + 2CH_3COONa$$

Iodide (I^-)

Layer test : On treating salt with dilute sulphuric acid, chloroform or carbon tetrachloride and chlorine water, gives violet coloured layer.

Chlorine replaces iodine that dissolves in chloroform.

$$\underset{\text{Salt}}{2NaI} + Cl_2 \longrightarrow 2NaCl + I_2$$

$$I_2 + \text{Chloroform} \longrightarrow \text{Violet coloured layer}$$

Starch paper test : Violet vapours with starch paper give blue colour.

$$I_2 + \text{Starch} \longrightarrow \underset{\text{Blue colour}}{I_2\text{-starch complex}}$$

Bromide (Br^-)

Layer test : On treating salt with dilute sulphuric acid, chloroform or carbon tetrachloride and chlorine water gives brown coloured layer.

Chlorine replaces bromine that dissolves in chloroform.

$$\underset{\text{Salt}}{2NaBr} + Cl_2 \longrightarrow 2NaCl + Br_2$$

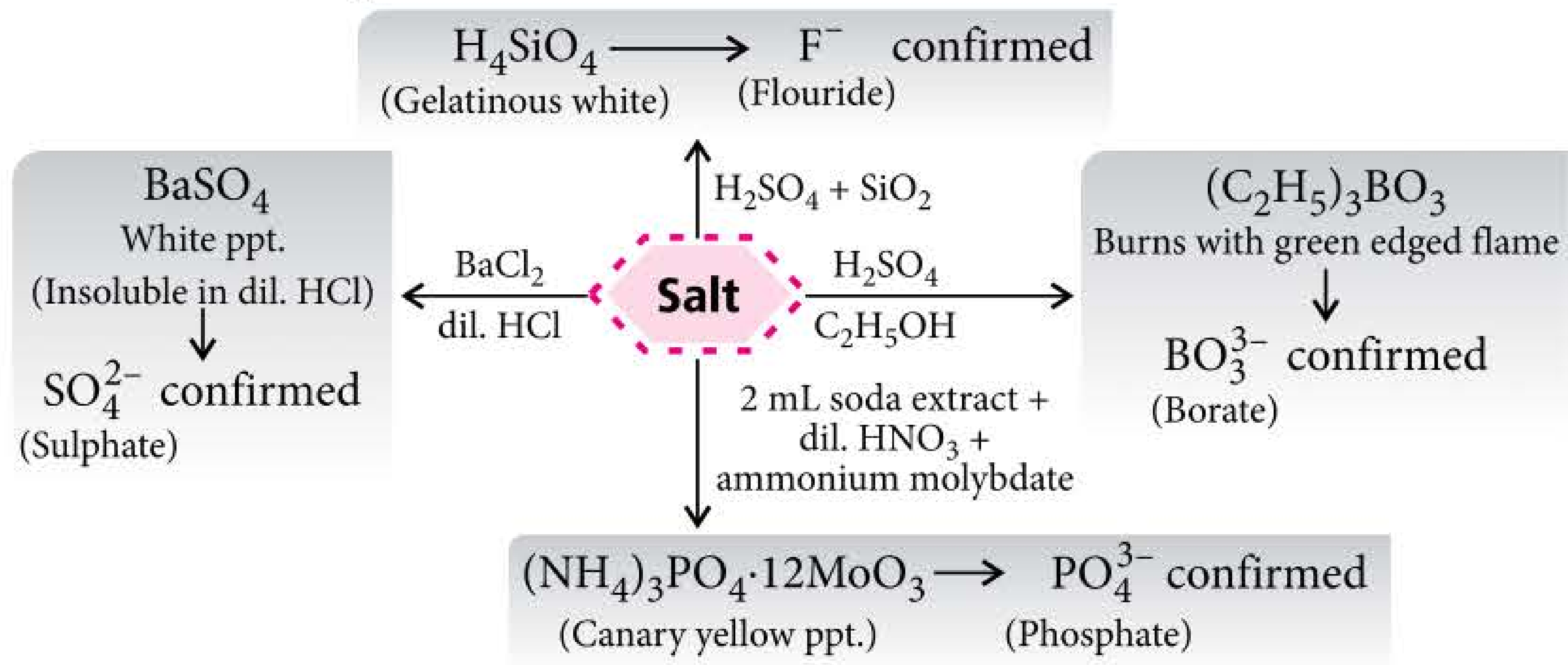
$$Br_2 + \text{Chloroform} \longrightarrow \text{Brown coloured layer}$$

INFO SHOTS

New system to detect mercury in water systems!

A new ultra-sensitive, low-cost and portable system for detecting mercury in environmental water has been developed by University of Adelaide researchers. The researchers team has engineered a nanoporous material called nanoporous anodic alumina to make a special structure called a rugate filter. The surface of the filter has been modified to make it selective to mercury ions. As water flows through the pores of the filter, the mercury ions become attached to the surface. An optical system–reflection spectroscopy–measures the amount of mercury present. A range of tests have shown the sensor can detect mercury at levels of 200 parts per billion in a complex mixture of other metal ions and environmental samples. Continued work will seek to enhance the optical signals for even higher sensitivity. The promising sensing performance of this system along with its cost-competiveness and portability make it an excellent potential alternative to current analytical techniques. This technique could provide the basis for future point-of-analysis systems for monitoring water quality on site and may help implement better monitoring processes around the world.

- **Third group :** These radicals cannot be detected by either dil. H_2SO_4 or conc. H_2SO_4. For detection of these acidic radicals we need some specific tests.



Cations or Basic Radicals

Group	Group reagent	Cations	Form of ppt.
I	dil. HCl	Pb^{2+}, Ag^+, Hg_2^{2+}	Chlorides
II	dil. HCl + H_2S gas	Pb^{2+}, Hg^{2+}, Cu^{2+}, Cd^{2+}, Bi^{3+}, Sb^{3+}, As^{3+}, Sn^{2+}/Sn^{4+}	Sulphides
III	$NH_4Cl + NH_4OH$	Fe^{3+}, Al^{3+}, Cr^{3+}	Hydroxides
IV	$NH_4Cl + NH_4OH + H_2S$ gas	Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}	Sulphides
V	$(NH_4)_2CO_3 + NH_4OH$	Ca^{2+}, Sr^{2+}, Ba^{2+}	Carbonates
VI	$Na_2HPO_4 + NH_4OH$	Mg^{2+}	–

- **Identification of basic radicals :**