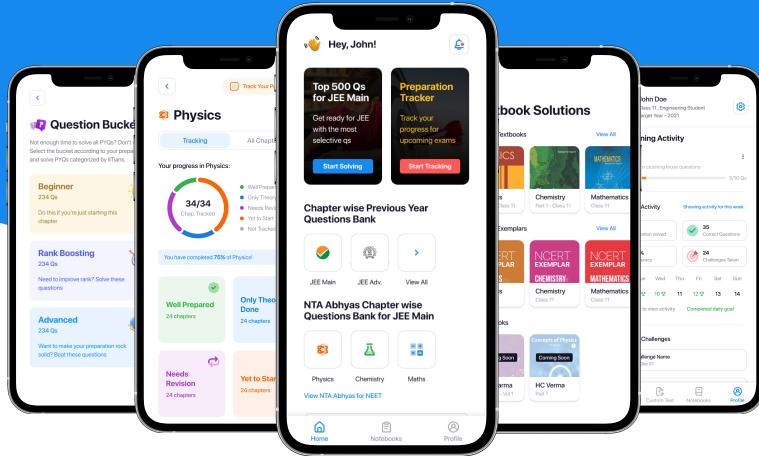




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Qualitative Analysis

Qualitative Inorganic Analysis

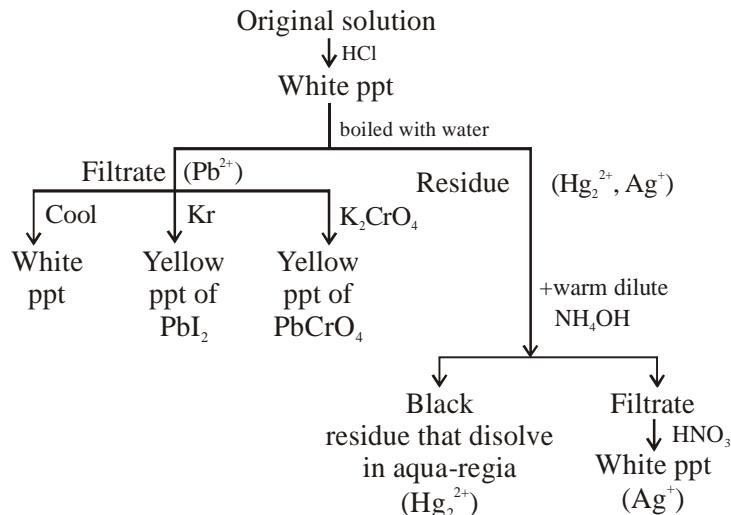
Qualitative inorganic analysis involves the detection of cations (basic radicals) and anions (acid radicals) present in an inorganic compound or a mixture of inorganic compounds. For a systematic study, qualitative analysis may be studied in the following parts :

- (A) Dry tests for inorganic compounds
- (B) Confirmatory tests for basic radicals
- (C) Confirmatory tests for acid radicals

Classification of Basic Radicals

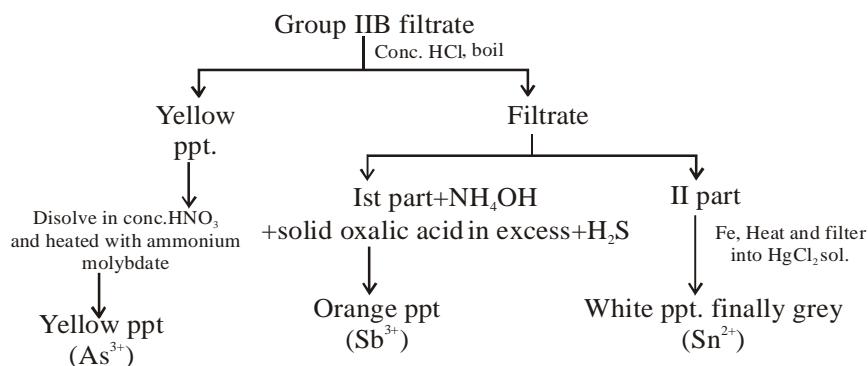
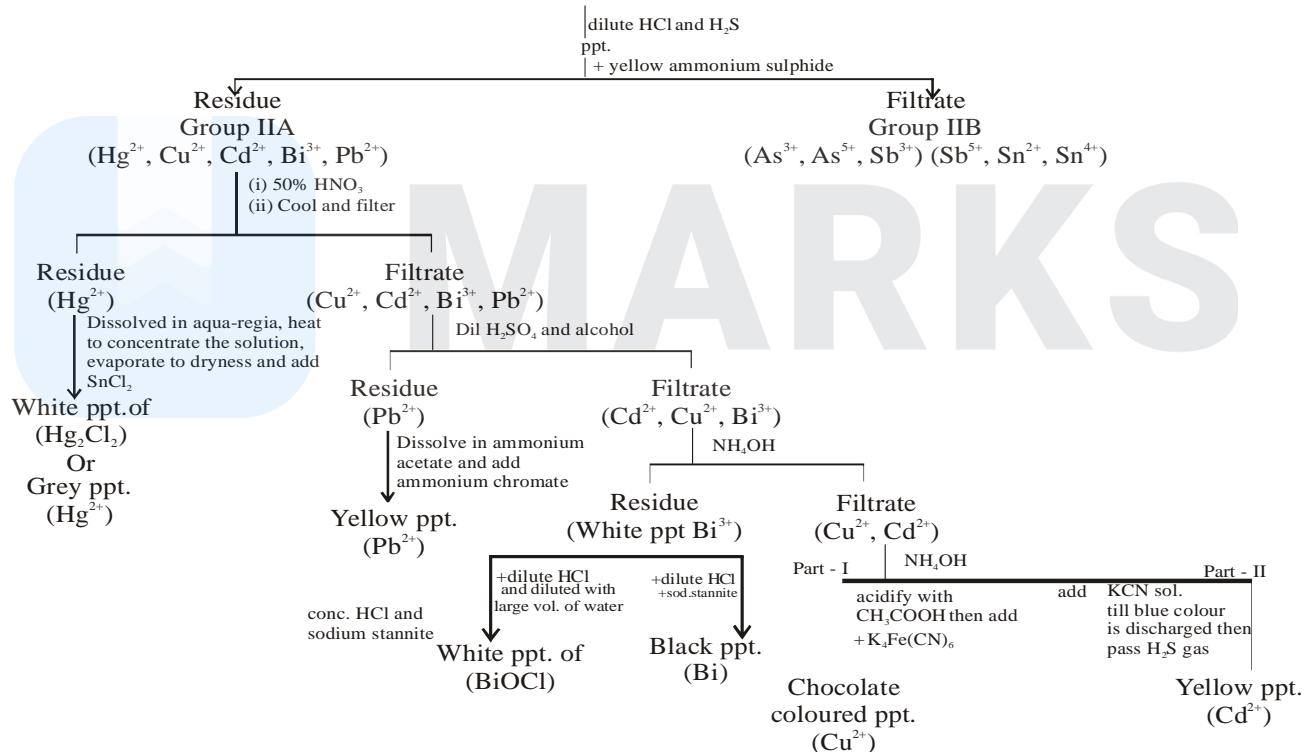
The qualitative analysis of basic radicals is based on the concept of solubility product, solubility and common ions. These concepts play very important role even in the classification of radicals in six groups and their group reagents. The common occurring cations have been divided into six groups.

Group	Group reagent	Basic radical	Composition and colour of the ppt.
I.	Dil. HCl	Ag^+ Pb^{2+} Hg_2^{2+}	AgCl White PbCl_2 White Hg_2Cl_2 White
II.A	H_2S in the presence of dil. HCl	Hg^{2+} Pb^{2+} Bi^{3+} Cu^{2+} Cd^{2+}	HgS Black PbS Black Bi_2S_3 Black CuS Black CdS Yellow
II.B	H_2S in the presence of dil. HCl	As^{3+} Sn^{4+} Sb^{3+} Sn^{2+}	As_2S_3 Yellow SnS_2 Yellow Sb_2S_3 Orange SnS Brown
III.	NH_4OH in the presence of excess of NH_4Cl	Fe^{3+} Cr^{3+} Al^{3+}	Fe(OH)_3 Reddish brown Cr(OH)_3 Dirty green Al(OH)_3 White gelatinous ppt.
IV.	H_2S in presence of NH_4OH	Co^{2+} Ni^{2+} Zn^{2+} Mn^{2+}	CoS Black NiS Black ZnS Bluish white MnS Buff (flesh) coloured
V.	$(\text{NH}_4)_2\text{CO}_3$ in the presence of NH_4OH	Ba^{2+} Sr^{2+} Ca^{2+}	BaCO_3 White SrCO_3 White CaCO_3 White
VI.	No specific group reagent	Mg^{2+} Na^+ K^+	$\text{Mg}(\text{NH}_4)\text{PO}_4$ White
Zero	NaOH	NH_4^+	NH_3 gas is evolved

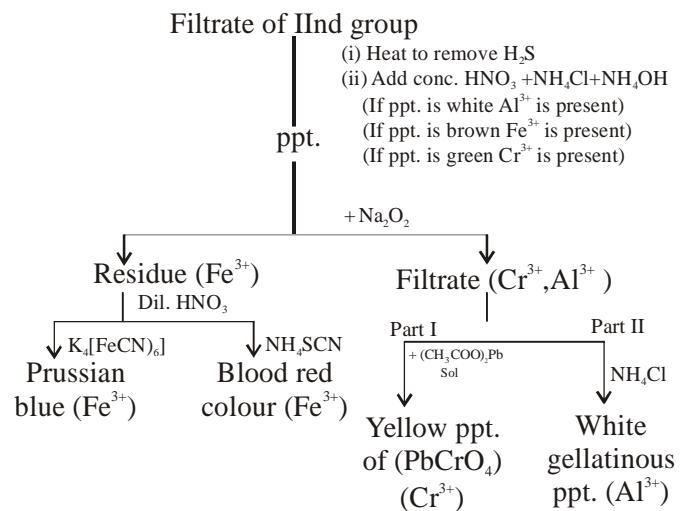
1. Ist Group Basic Radicals : $(\text{Ag}^+, \text{Pb}^{2+}, \text{Hg}_2^{2+})$ 

2. IIInd Group Basic Radicals : Original solution or Ist group filtrate

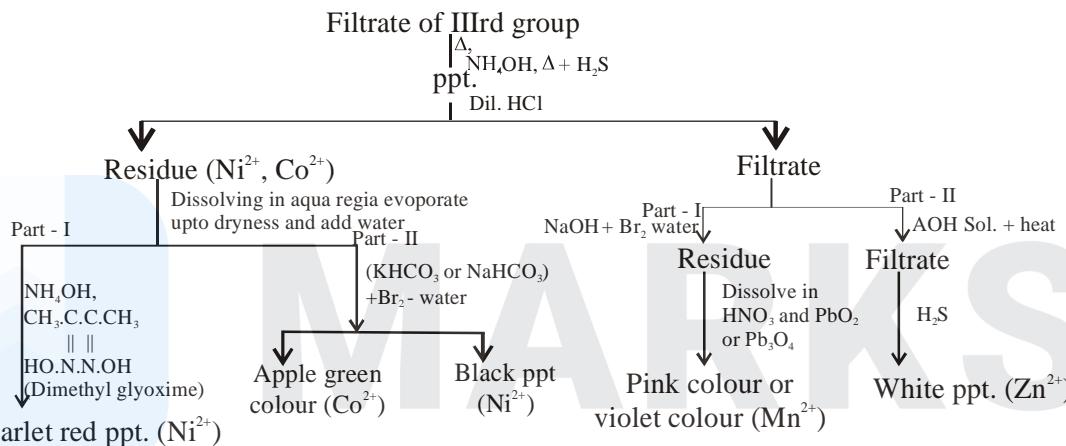
Original solution or Ist group filtrate
(If Ist group is absent)



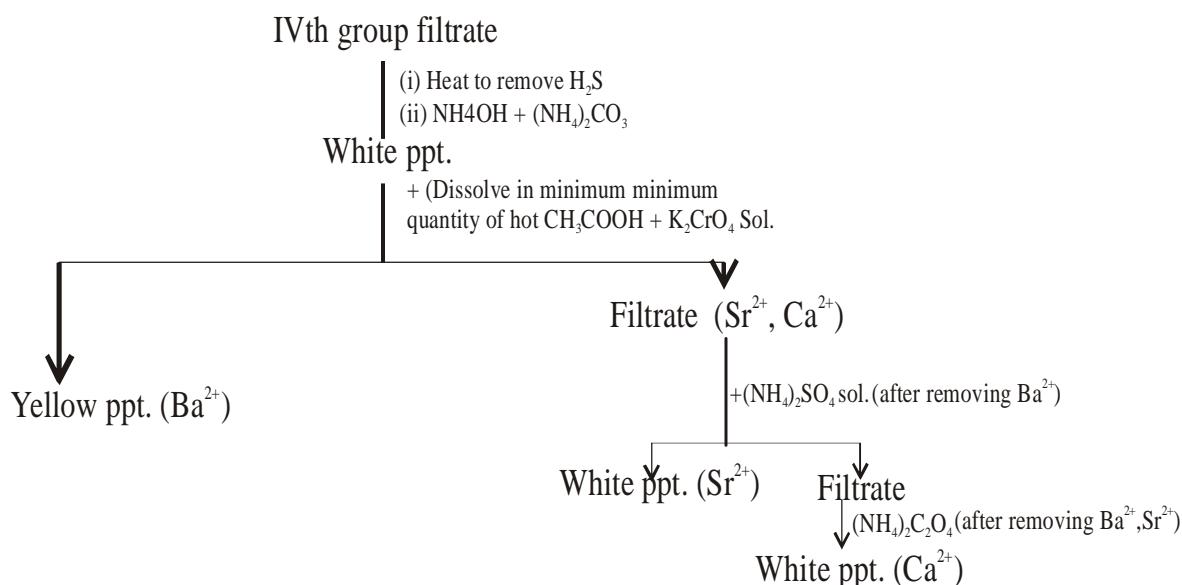
IIIrd Group Basic Radicals :



4. IV Group Basic Radicals : (Ni^{2+} , Co^{2+} , Mn^{2+} , Zn^{2+}) :



5. Vth Group Basic Radicals : (Ba^{2+} , Sr^{2+} , Ca^{2+})



6. VI Group Basic Radical

Filtrate of Vth group

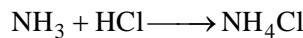
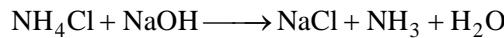
↓
White crystalline ppt.

Heat and add $(\text{NH}_4)_2\text{C}_2\text{O}_4$. If any ppt. is obtained. Now add NH_4OH and Na_2HPO_4 scratch with glass rod.

7. Zero Group Basic Radical : It contains NH_4^+ ion.

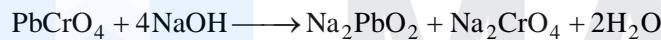
- (a) The salt is heated with NaOH , ammonia is evolved. The ammonia gives white fumes with HCl .
- (b) NH_4^+ gives brown ppt. with Nessler's reagent.

Reactions :

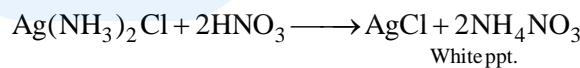
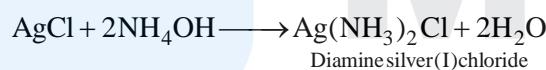


Reaction involve :

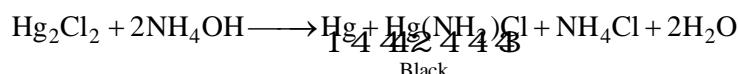
Pb^{2+}



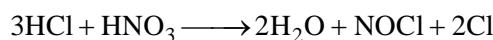
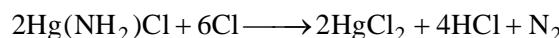
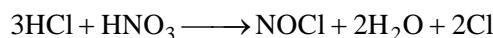
Ag^+



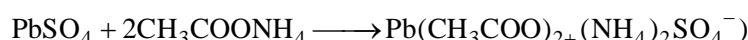
Hg_2^{2+}

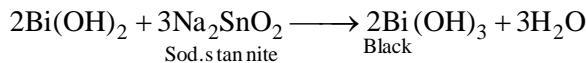
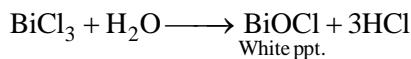
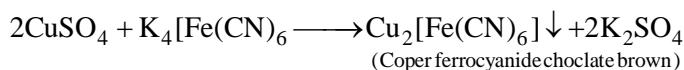
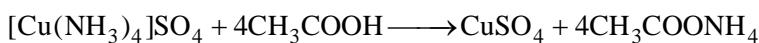
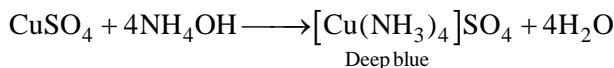
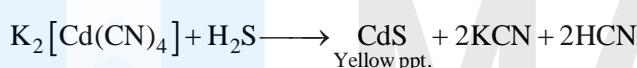
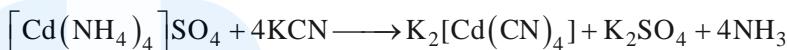
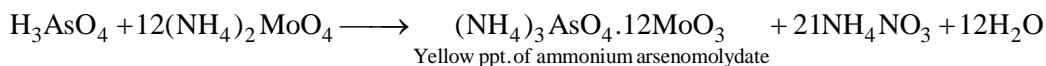
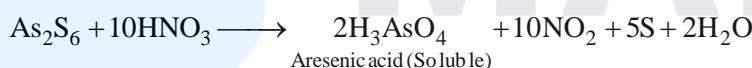
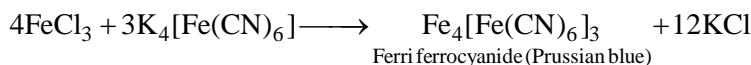
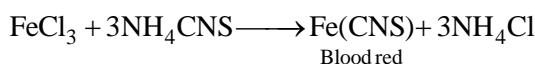
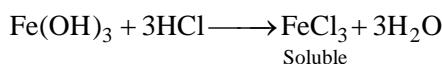
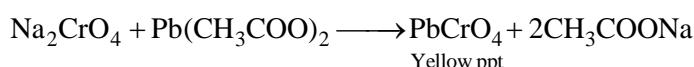
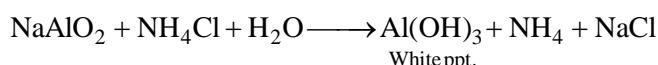


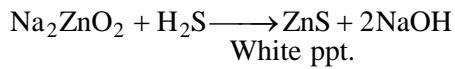
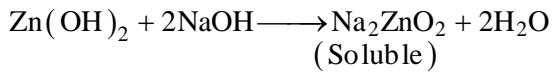
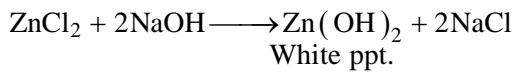
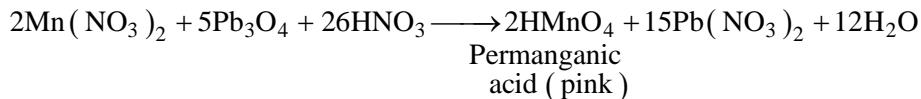
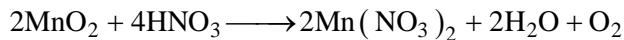
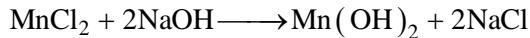
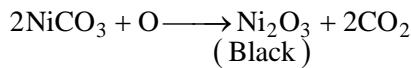
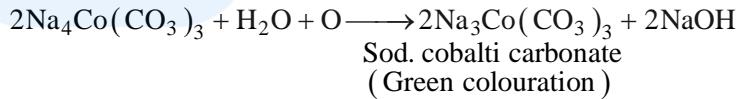
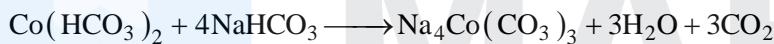
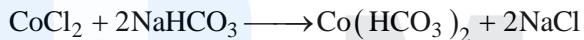
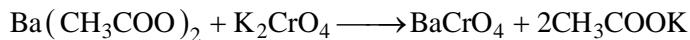
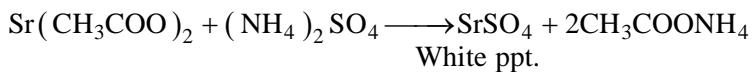
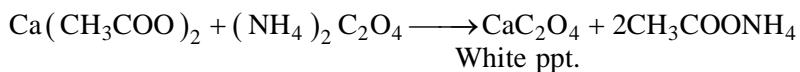
Hg^{2+}



Pb^{2+}



Bi^{3+}  Cu^{2+}  Cd^{2+}  As^{3+}  Ie^{3+}  Cr^{3+}  Al^{3+} 

Zn^{2+}  Mn^{2+}  Ni^{2+}  Ba^{2+} (barium) Sr^{2+} (strontium) Ca^{2+} (calcium)

DRY TEST

Dry tests are of great importance as these tests give clear indications of the presence of certain radicals. The following tests are performed in dry state:

- (i) Flame test
- (ii) Borax bead test
- (iii) Micro-cosmic salt bead test
- (iv) Charcoal cavity test
- (v) Cobalt nitrate charcoal test

Flame Test

Alkali and Alkaline earth Metals Salts give characteristic colour when introduced in Bunsen flame. The colour produced by them are given below.

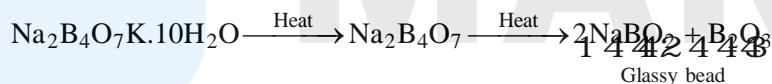
Li	Grimson red	Ca	Brick red
Na	Golden yellow	Sr	Crimson
K	Violet	Ba	Apple green

Note :

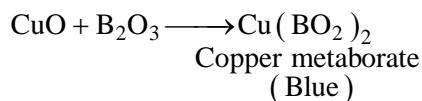
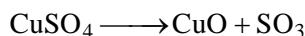
Flame test should not be performed in the presence of As, Sb, Bi, Sn and Pb as these radicals form alloy with platinum and hence, the wire is spoiled.

Borax bead test

On heating borax the colourless glassy bead formed consists of sodium metaborate and boric anhydride.



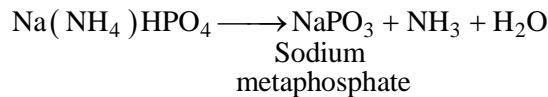
On heating with a coloured salt, the glassy bead forms a coloured metaborate in oxidising flame.



Metal	Colour of the bead in			
	Oxidising-flame		Reducing-flame	
	Hot	Cold	Hot	Cold
Copper	Green	Blue	Colourless	Brown-red
Iron	Brown-yellow	Pale-yellow	Bottle green	Bottle green
Chromium	Green	Green	Green	Green
Cobalt	Blue	Blue	Blue	Blue
Manganese	Violet	Amethyst red	Grey	Grey
Nickel	Violet	Brown	Grey	Grey

Microcosmic salt bead test

This test is similar to borax bead test. When microcosmic salt is heated, a colourless transparent bead of sodium metaphosphate is formed.



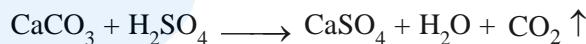
Sodium metaphosphate combines with metallic oxides to form orthophosphates which are usually coloured. The shade of the colour gives a clue regarding the presence of metal.

Metal	Colour of the bead in			
	Oxidising-flame		Reducing-flame	
	Hot	Cold	Hot	Cold
Copper	Green	Blue	Colourless	Red
Iron	Yellow or reddish-brown	Yellow	Yellow	Colourless
Chromium	Green	Green	Green	Green
Manganese	Violet	Violet	Colourless	Colourless
Cobalt	Blue	Blue	Blue	Blue
Nickel	Brown	Brown		Grey

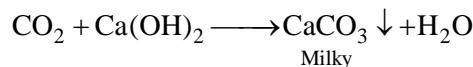
Test for acid radicals : Add dil. HCl or H_2SO_4 to a small amount of substance and warm gently, observe.

Carbonate or CO_3^{2-} :

- (i) Brisk effervescence of colourless gas CO_2 ; may be carbonate.



- (ii) The gas turns lime water milky

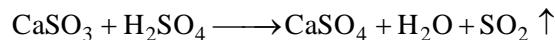


- (iii) Excess of passage of gas through lime water is milky ness disappears

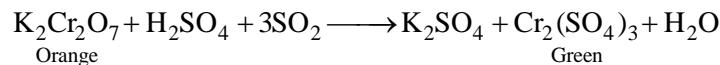


Sulphite or SO_3^{2-} :

- (i) Colourless gas with suffocating odour of burning sulphur; may be sulphite.



- (ii) The gas turns orange colour of acidic $K_2Cr_2O_7$ to green



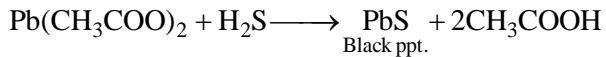
Note : CO_2 does not turn orange colour of acidic $\text{K}_2\text{Cr}_2\text{O}_7$ to green

Sulphide (S^{2-}) :

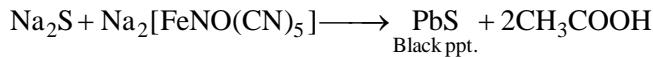
- (i) The sulphide salts from H_2S which smells like rotten eggs.



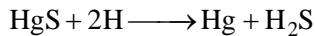
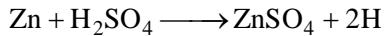
- (ii) On exposure to this gas, the lead acetate paper turns black due to the formation of lead sulphide.



- (iii) The sulphides also turn sodium nitroprusside solution violet (use sodium carbonate extract for this test).

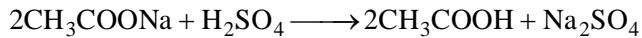


Note : Sulphide of lead, calcium, nickel, cobalt, antimony and stannic are not decomposed with dil. H_2SO_4 . Conc. HCl should be used for their test. However, brisk evolution of H_2S takes place even by use of dil. H_2SO_4 if a pinch of zinc dust is added.



Acetate (CH_3COO^-)

- (i) Acetates decompose to give acetic acid vapours having characteristic smell of vinegar.



- (ii) All acetates are soluble in water and their aqueous solution on addition to neutral FeCl_3 solution develops a blood red colour due to the formation of ferric acetate.



Note :

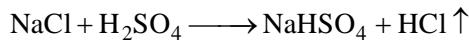
1. The ferric chloride solution supplied in the laboratory is always acidic due to hydrolysis. It is made neutral by the addition of dil. solution of NH_4OH drop by drop with constant stirring till the precipitate formed does not dissolve. The filtrate is called neutral ferric chloride solution.
2. Before testing acetate in the aqueous solution by FeCl_3 , it must be made sure that the solution does not contain,



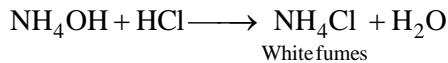
since these also combine with Fe^{+3} . Therefore the test of acetate should be performed by neutral ferric chloride solution only after the removal of these ions by AgNO_3 solution.

Chloride (Cl^-)

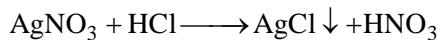
- (i) Colourless pungent fumes of hydrogen chloride are evolved.



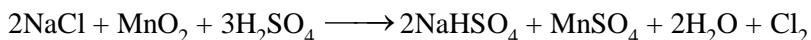
- (ii) The gas evolved forms white fumes of ammonium chloride with NH_4OH .



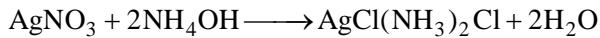
- (iii) The gas evolved or solution of chloride salt forms a curdy precipitate of silver chloride with silver nitrate solution.



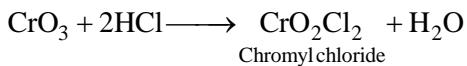
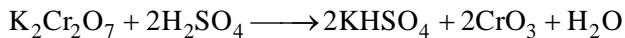
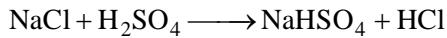
- (iv) Yellowish-green chlorine gas with suffocating odour is evolved on addition of MnO_2 to the above reaction mixture.



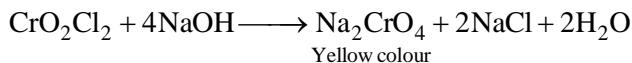
Note : The curdy precipitate of AgCl dissolves in ammonium hydroxide forming a complex salt.



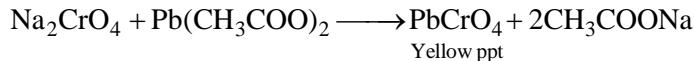
(v) Chromyl chloride test : When solid chloride is heated with conc. H_2SO_4 in presence of $\text{K}_2\text{Cr}_2\text{O}_7$, deep red vapours of chromyl chloride are evolved.



These vapours on passing through NaOH solution, form the yellow solution due to the formation of sodium chromate.



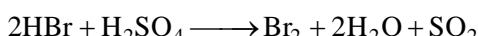
The yellow solution neutralized with acetic acid gives a yellow solution due to the formation of sodium chromate.



Note :

1. This test is not given by the chloride of mercury, tin, silver lead and antimony.
 2. The chromyl chloride test is always to be performed in a dry test tube otherwise the chromyl chloride vapours will be hydrolysed in the test tube.
 3. Bromides and iodides do not give this test.
2. Bromide (Br^-)

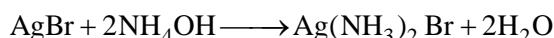
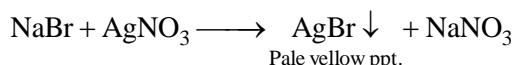
- (i) Reddish-brown fumes of bromine are formed.



- (ii) More reddish-brown fumes of bromine are evolved when MnO_2 is added

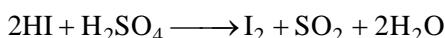


- (iii) The aqueous solution of bromide or sodium carbonate extract gives pale yellow precipitate of silver bromide which partly dissolves in excess of NH_4OH forming a soluble complex.



3. Iodide (I^-)

- (i) Violet vapours of iodine are evolved.



(ii) Violet vapours with starch produce blue colour.

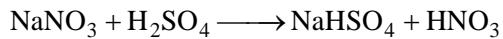


(iii) More violet vapours are evolved when MnO_2 is added.



4. Nitrate (NO_3^-)

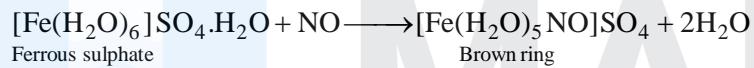
(i) Light brown fumes of nitrogen dioxide are evolved.



(ii) These fumes intensify when copper turnings are added.

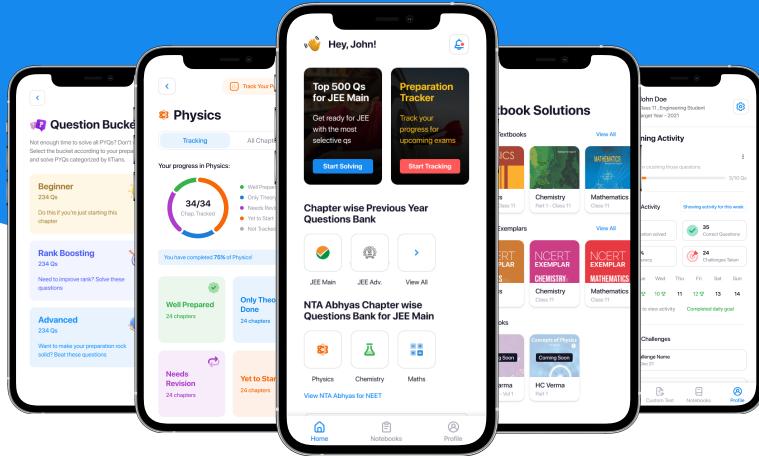


(iii) Brown ring test : An aqueous solution of salt containing nitrate is mixed with freshly solution of salt solution and conc. H_2SO_4 is poured in test tube from sides, a brown ring is formed an account of the formation of a complex at the junction of two liquids.





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