

# Qualitative Inorganic Analysis

The investigation and recognition of substances is a very important branch of chemistry, and the skill of the analyst is essential today in medicine, criminology and the manufacture of countless products.

Qualitative inorganic analysis is concerned with the identification of radicals present in an inorganic salt or a mixture of salts. Simple tests are available for showing the presence of these radicals. The tests are carried out in right order.

Inorganic salts are formed when acids react with bases. Each inorganic salt consists of two parts, i.e. part contributed by acid is called acidic radical or anion and the part contributed by base is called basic radical or cation. For example, in sodium chloride.  $\text{Na}^+$  is basic radical or cation and  $\text{Cl}^-$  is acidic radical or anion

Before you start the test of an inorganic substance always note the appearance of the substance (colour, state), and determine if it is soluble in water. If it is, test with litmus to see if the solution is acidic, alkaline or neutral. Colour and solubility of inorganic substance in water may help to identify some basic and acidic radicals.

Table 7.1 Colour of some inorganic salts

Colour	Compounds
White or colourless	Salts of Na, K, NH, Mg, Ca, Sr, Ba, Zn, and Al
Blue	Hydrated cupric salt and anhydrous cobalt salt
Pink	Hydrated salt of Co and Mn
Green	Fe (ous) salt, except $\text{FeS}$ , $\text{CuCl}_2$ , $2\text{H}_2\text{O}$ , $\text{CuCO}_3$ N (ous) salt
Black	$\text{FeS}$ , $\text{CoS}$ , $\text{NiS}$ , $\text{PbS}$

Table 7.2 Solubility of inorganic compounds in water

Compounds	Solubility in Water
Nitrates	All salts are soluble
Sulphates	All except Pb, Fe(ic), Ba, Sr and Ca
Nitrites	All except Ag
Chlorides Bromides and iodides	All Chlorides Bromides and iodides except and
Carbonates	Na, K and NH: only
Sulphate	Na, K and NH: only
	Na, K and NH: only

The qualitative analysis of an inorganic substance is generally carried out in the following four parts

1. Dry test for Basic Radicals
2. Dry test for Acidic Radicals
3. Wet test for anions(acidic radicals)
4. Wet test for anions(acidic radicals)

**Aim:** To determine acid and basic radical in a given salt sample.

**Physical characteristic:**

(1) colour -

(2) odour -

(3) solubility -

(4) physical appearance -



### Dry tests for basic radicals

#### Action of heat

Perform this experiment by heating a small amount of salt in a dry test tube.

Information from this test can give valuable clues, partly from observation of the residue partly from identification of any gas evolved. Tabulate your observation along with the inferences that you can draw

Experiment	Observation	Inference
heat a pinch of the solid in a dry test tube	<p>1) (Residue) Change in colour</p> <ul style="list-style-type: none"> <li>i. Yellow when hot, white when cold</li> <li>ii. Yellow in both hot and cold conditions</li> <li>iii. Red while hot and yellow or brown when cold.</li> <li>iv. Black in both and cold conditions</li> </ul> <p>2) Salt fuses <i>melting</i></p> <p>3) Decrepitation</p> <p>The salt decrepitates (flies to pieces with sharp crackling sound)</p> <p>4) Formation of sublimate (<i>Fog like Formation</i>) White sublimate formed</p> <p>5) Evolution of gases</p> <ul style="list-style-type: none"> <li>i. Colourless and odourless gas, turns lime water milky</li> <li>ii. Evolution of ammonia-gives white fumes when a glass rod dipped in dil HCl is brought near the mouth of the test tube</li> </ul>	<p>Zn<sup>2+</sup> Pb<sup>2+</sup> Fe<sup>3+</sup></p> <p>salt of Cu<sup>2+</sup>, Co<sup>2+</sup>, Mn</p> <p>Na<sup>+</sup>, K<sup>+</sup>, Ca<sup>2+</sup>, Sr<sup>2+</sup>, Ba<sup>2+</sup></p> <p>Pb(NO<sub>3</sub>)<sub>2</sub>, NaCl</p> <p>NH<sub>4</sub><sup>+</sup></p> <p>CO<sub>3</sub><sup>2-</sup> or C<sub>2</sub>O<sub>4</sub><sup>2-</sup> present</p> <p>NH<sub>4</sub><sup>+</sup></p>

## (B) Dry tests for acid radicals

Dry tests for acid radicals are performed in the following manner

- 1) Action of diluted hydrochloric acid
- 2) Action of concentrated sulphuric acid

### 1. Action of dil. hydrochloric acid

Experiment	Observation	Inference
Take a small amount of the substance and add 5 ml of dil hydrochloric acid, If no reaction takes place, warm a little	<ul style="list-style-type: none"> <li>i. Colourless and odourless gas is evolved with effervescence, turns lime water milky.</li> <li>ii. Colourless gas with odour of rotten eggs turns lead acetate paper black.</li> <li>iii. Colourless gas possessing suffocating odour, turns acidified potassium dichromate solution green</li> <li>iv. Reddish-brown gas is evolved turns acidified Starch-iodide paper blue (starch-iodide paper is prepared by moistening a piece of filter paper with a KI solution and then with a starch solution).</li> <li>v. Colourless gas with smell of vinegar</li> </ul>	<ul style="list-style-type: none"> <li>Carbonate(<math>\text{CO}_3^{2-}</math>) and Bicarbonate(<math>\text{HCO}_3^-</math>)</li> <li>Sulphate(<math>\text{S}^{2-}</math>)</li> <li>Sulphite(<math>\text{SO}_3^{2-}</math>)</li> <li>Nitrite(<math>\text{NO}_2^-</math>)</li> <li>Acetate(<math>\text{CH}_3\text{COO}^-</math>)</li> </ul>

### 2. Action of concentrated sulphuric acid

Experiment	Observation	Inference
1) Take a small amount of the substance in a dry test tube. Add 4-5 ml, of concentrated $\text{H}_2\text{SO}_4$ , and gently heat the solution.	<ul style="list-style-type: none"> <li>i. Colourless gas with pungent smell gives white fumes when glass rod moistened with <math>\text{NH}_4\text{OH}</math> is held near the mouth of the test tube.</li> <li>ii. Reddish brown gas is evolved.</li> <li>iii. Violet vapours are evolved turns starch paper blue</li> <li>iv. Browns fumes evolved.</li> </ul>	<ul style="list-style-type: none"> <li>Chloride (<math>\text{Cl}^-</math>)</li> <li>Bromide(<math>\text{Br}^-</math>)</li> <li>Iodide(<math>\text{I}^-</math>)</li> <li>Nitrate(<math>\text{NO}_3^-</math>)</li> </ul>

2) Take small amount of substance in a dry test tube. Add a little $MnO_2$ and 2 ml. conc. $H_2SO_4$ . Heat gently	i. Greenish-yellow gas turns moist starch-iodide paper blue. ii. Brown-red vapours turn starch-iodide paper blue. iii. Violet vapours turns starch paper blue.	Chloride ( $Cl^-$ ) Bromide ( $Br^-$ ) Iodide ( $I^-$ )
3) Take a small amount of the substance in a dry test tube. Add 3-4 ml conc. $H_2SO_4$ , and a little Cu turning. Heat gently	Reddish-brown fumes and the solution in the test tube appear blue.	Nitrate ( $NO_3^-$ )

(C)

**Wet test for acid radical**If salt not soluble in  $H_2O$  then prepare  $Na_2CO_3$  extract. Otherwise water extract

Experiment	Observation	Inference
1. Take about 5 ml of $H_2O$ extract/ $Na_2CO_3$ extract in a test tube. Acidify with dil. HCl. Boil to expel CO. Add $BaCl_2$ solution.	White ppt insoluble in dil HCl or dil $HNO_3$	Sulphate ( $SO_4^{2-}$ )
2. Take 5 ml of sodium carbonate extract in a test tube. Acidify with dil. HCl. Add $BaCl_2$ solution followed by bromine water. Heat the content	White ppt.	Sulphite ( $SO_3^{2-}$ )
3. Take 5 ml of sodium carbonate extract in a test tube. Acidify with dil $H_2SO_4$ . Add about 5 ml of freshly prepared $FeSO_4$ solution. Slowly pour 2-3 ml of conc. $H_2SO_4$ down the side of the tube. Hold the	A brown ring at the junction of the two liquids.	Nitrate ( $NO_3^-$ )

<p>water extract</p> <p>tube at an angle while doing this. The heavy Sulphuric acid sinks to the bottom.</p> <p>4. Take 5ml of <math>\text{Na}_2\text{CO}_3</math> extract in a test tube. Acidify with dil <math>\text{HNO}_3</math> Boil off <math>\text{CO}_2</math> Add <math>\text{AgNO}_3</math> solution.</p>	<p>White ppt. readily soluble in <math>\text{NH}_4\text{OH}</math>. Or Pale yellow ppt. soluble in conc <math>\text{NH}_4\text{OH}</math> solution. Or Yellow ppt. insoluble in conc <math>\text{NH}_4\text{OH}</math></p>	<p><b>Chloride (<math>\text{Cl}^-</math>)</b> <b>Bromide (<math>\text{Br}^-</math>)</b> <b>Iodide (<math>\text{I}^-</math>)</b></p>
<p>water extract</p> <p>5. Take 5 ml of <math>\text{Na}_2\text{CO}_3</math> extract. Acidify with acetic acid and then add lead acetate solution.</p>	<p>Black ppt soluble in hot dil <math>\text{HNO}_3</math>.</p>	<p><b>Sulphide (<math>\text{S}^{2-}</math>)</b></p>
<p>water extract</p> <p>6. Take 5 ml of <math>\text{Na}_2\text{CO}_3</math> extract; add a few drops of sodium nitro prusside solution.</p>	<p>Violet or Pink colouration</p>	<p><b>Sulphide (<math>\text{S}^{2-}</math>)</b></p>
<p>water extract</p> <p>7. Take 2-3 ml <math>\text{Na}_2\text{CO}_3</math> extract in a test tube. Acidify with dil <math>\text{H}_2\text{SO}_4</math> Then add <math>\text{KI}</math> solution.</p>	<p>Reddish-brown colouration, turns starch solution blue.</p>	<p><b>Nitrate (<math>\text{NO}_3^-</math>)</b></p>
<p>water extract</p> <p>8. Take 5 ml of <math>\text{Na}_2\text{CO}_3</math> extract in a test tube Add 2-3 ml of conc. <math>\text{HNO}_3</math> and 10 ml of ammonium molybdate solution Warm moderately (<math>40^\circ\text{C}</math>)</p>	<p>Straw-coloured ppt (light yellow)</p>	<p><b>Phosphate (<math>\text{PO}_4^{3-}</math>)</b></p>
<p>water extract</p> <p>9. Take 2-3 ml of <math>\text{Na}_2\text{CO}_3</math> extract in a test tube Acidify with dil acetic acid Add <math>\text{CaCl}_2</math> solution.</p>	<p>White ppt soluble in dilute <math>\text{HCl}</math></p>	<p><b>Oxalate (<math>\text{C}_2\text{O}_4^{2-}</math>)</b></p>
<p>water extract</p> <p>10. Dissolve salt in water: To the aqueous solution add freshly</p>	<p>Blood red colouration</p>	<p><b>Acetate (<math>\text{CH}_3\text{COO}</math>)</b></p>

prepared $\text{FeCl}_2$ solution.		
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## Wet test for basic radical

1. Distilled water (cold/hot)
2. Dil HCl (cold/hot)
3. Conc HCl (cold hot)

Solubility of inorganic salt in above solvents may often help to identify some basic radicals

Solvent	Salt
1. Cold water	a) All $\text{NH}_4$ , Na and K salts b) All nitrites, nitrates and acetates c) Most of the sulphates except those of Pb, Ba, Ca, Sr. d) All chlorides except that of lead
2. Hot water	Lead chloride, lead nitrate  All carbonates which do not dissolve in water, i.e. carbonates of Ca, Ba, Sr, Mg, Fe, etc but not of Pb

The usual procedures for analyzing a salt involve systematic separation of basic radical into group. The table given below gives briefly the classification of basic radicals into groups reagents and the form in which they are precipitated.

Group	Group Reagent	Basic Reagent	Form in which basic radicals are precipitated
I	Dil HCl	$\text{Pb}^{2+}$ , $\text{Hg}^{2+}$ (ous), $\text{Ag}^+$	Chlorides
II	$\text{H}_2\text{S}$ in presence of dil HCl	$\text{Pb}^{2+}$ , $\text{Cu}^{2+}$ , $\text{As}^{3+}$ , $\text{Hg}^{2+}$ , $\text{Cd}^{2+}$ , $\text{Bi}^{3+}$ , $\text{Sb}^{3+}$ , $\text{Sn}^{2+}$	Sulphides
III	$\text{NH}_4\text{OH}$ in presence of $\text{NH}_4\text{Cl}$	$\text{Fe}^{3+}$ , $\text{Al}^{3+}$ , $\text{Cr}^{3+}$	Hydroxides
IV	$\text{H}_2\text{S}$ in presence of $\text{NH}_4\text{OH}$	$\text{Ni}^{2+}$ , $\text{Mn}^{2+}$ , $\text{Zn}^{2+}$ , $\text{Co}^{2+}$	Sulphides
V	$(\text{NH}_4)_2\text{CO}_3$ in the presence of $\text{NH}_4\text{OH}$	$\text{Ca}^{2+}$ , $\text{Sr}^{2+}$ , $\text{Be}^{2+}$	Carbonates
VI	—	$\text{Mg}^{2+}$ , $\text{Na}, \text{K}$	—

(Metal ions in the box are not included in the syllabus)

Division of the basic radicals (cations) in different groups (group seperstion)

Experiments	Observation	Inference
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1. Add dilHCl to the salt solution.	White ppt.	Group I present.
2. Add dil HCl to the salt solution and then pass $H_2S$ gas.	Black ppt.	Group II present.
3. Boil salt solution with salt + HCl and add $NH_4Cl$ and $NH_4OH$ solution in excess.	(a)Reddish-brown ppt. (b)White gelatinous ppt.	Group III present (a)Fe salt (b)Al salt
4. Add $NH_4Cl$ and $NH_4OH$ to salt solution and pass $H_2S$ gas.	(a)Black ppt. (b)Pink ppt. (c)White ppt.	Group IV present (a)Co salt (b)Mn salt (c)Zn salt
5. Add $NH_4OH$ and $(NH_4)_2CO_3$ to the salt solution.	White ppt.	Group V present maybe Ca, Ba, Sr salt.
6. No particular group reagent.	_____	Group VI Mg salt.
7. Salt solution + Conc. NaOH	(a)An pungent smell (b)White fumes—becomes dense when glass rod dipped in dil. HCl is brought near the mouth.	

Group analysis:

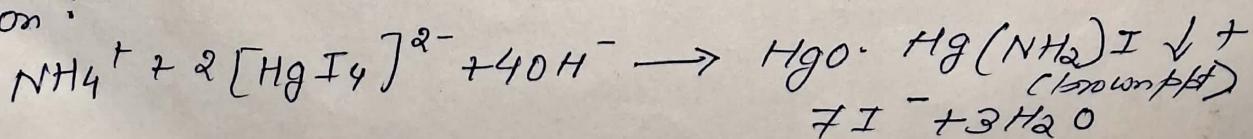
## Analysis of Group Zero :-

Experiment	OBSERVATION	INFERENCE
1. Heat salt + NaOH + Bring a glass rod dipped in dil HCl near the mouth of test tube	Dense white fumes	$NH_4^+$ present
2. Heat salt + NaOH + Nessler reagent	Brown ppt	$NH_4^+$ confirmed.

SWAPNANEEL DEY

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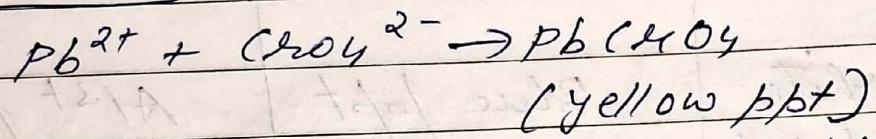
Reaction :



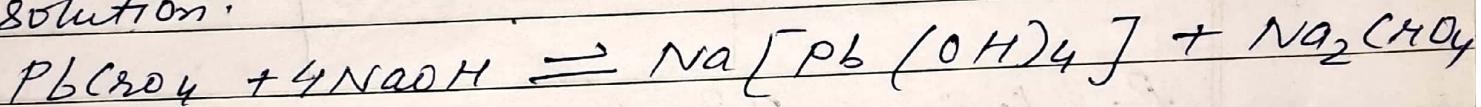
## Analyses of Group I.

EXP	OBS	INF
sample + few drops of $K_2CrO_4$	yellow ppt	$Pb^{2+}$ confirmed.

Reaction:



This yellow ppt is soluble in hot NaOH solution.

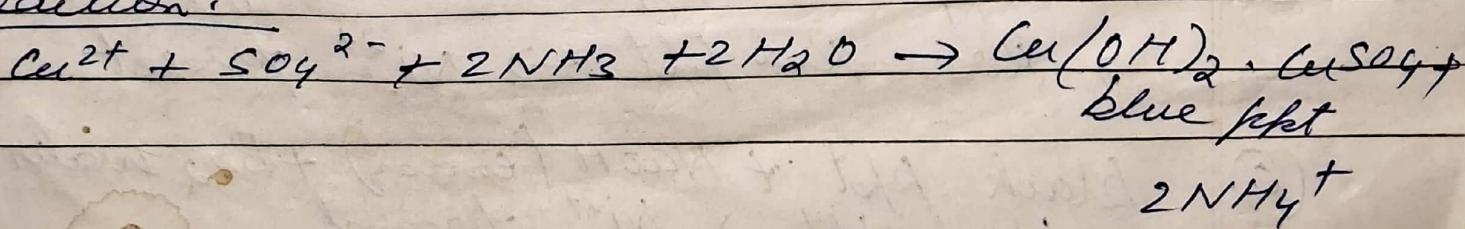


## Analysis of Group II

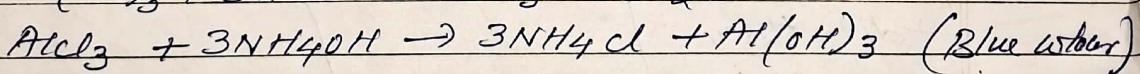
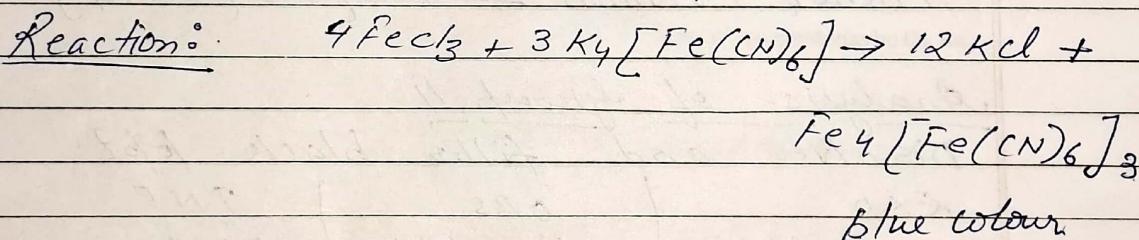
Dissolve and filter black ppt.

EXP	OBS	INF
Boil the black ppt + dil $HNO_3$ + dil $H_2SO_4$	white ppt is formed	$Pb^{2+}$ present
If no white ppt then add excess of $NH_4OH$ solution to the solution(1)	deep blue colour is observed	$Cu^{2+}$ present

Reaction:



<u>EXPERIMENT</u>	<u>OBSERVATION</u>	<u>INFERENCE</u>	
(1) Filter the <sup>brown</sup> ppt and dissolve the ppt in dil HCl and add Potassium ferrocyanide test	blue colour	$\text{Fe}^{3+}$ present	(1)
(2) Dissolve white ppt in dil HCl + $\text{NH}_4\text{OH}$	Blue ppt	$\text{Al}^{3+}$ present	(2)



#### Analysis of Group IV

(a) Filter the ppt (<sup>black ppt</sup>) and add  $\text{NH}_4\text{OH}$  and add acetic acid and potassium nitroite — yellow ppt —  $\text{Co}^{2+}$  present

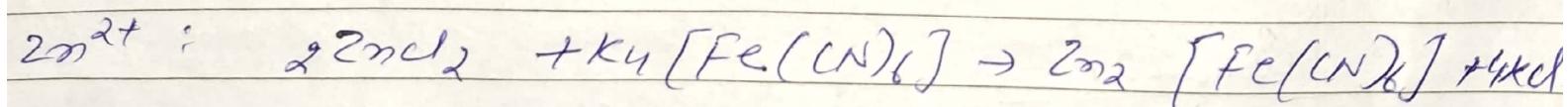
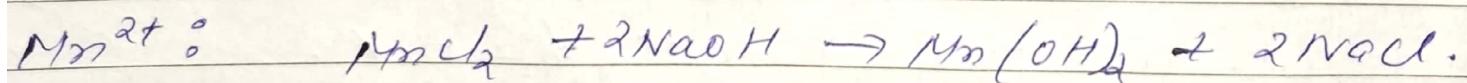
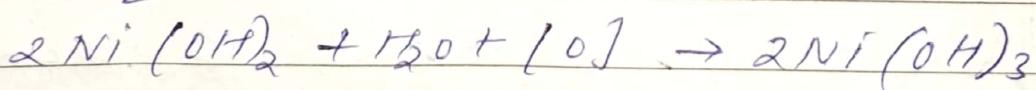
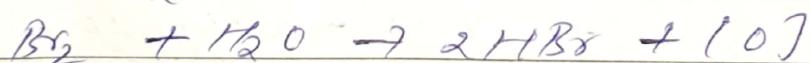
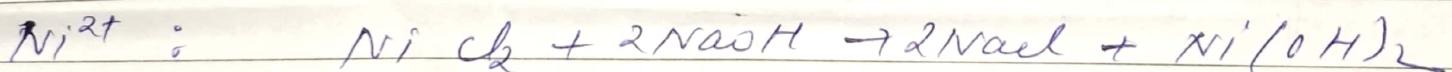
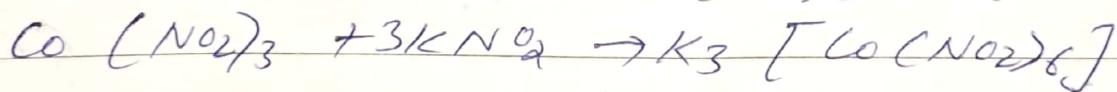
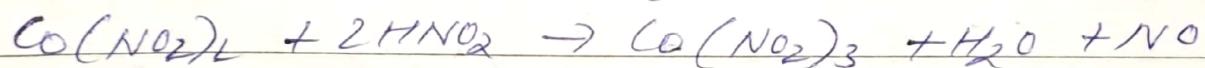
(b) black ppt +  $\text{NH}_4\text{OH}$  + dimethyl/ethyl glyoxime  
 $\rightarrow$  Rose Red ppt  $\rightarrow \text{Ni}^{2+}$  present

(c) black ppt +  $\text{NaOH}$  (excess) +  $\text{Br}_2$  water + boil  $\rightarrow$   
 black ppt  $\rightarrow \text{Ni}^{2+}$  present

flesh coloured ppt + add NaOH + shake + white ppt is formed + add  $\text{Br}_2$  water to white ppt  $\rightarrow$  It turns black or brown  $\rightarrow \text{Mn}^{2+}$  present.

Fall white ppt + NaOH + white ppt is formed + NaOH + white ppt dissolves  $\rightarrow \text{Zn}^{2+}$  present.

Small white ppt + hot ferrocyanide soln + white or bluish white ppt is formed  $\rightarrow \text{Zn}^{2+}$  present.

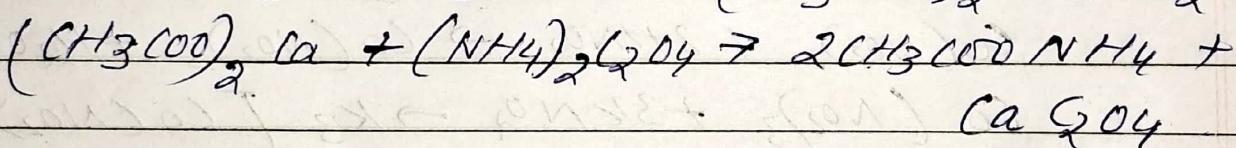
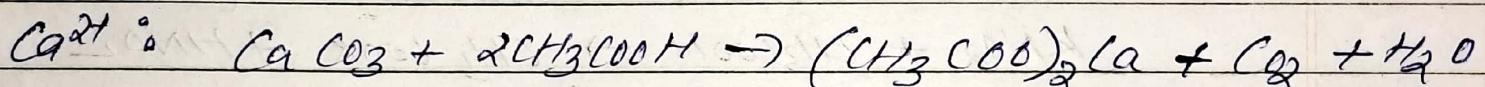
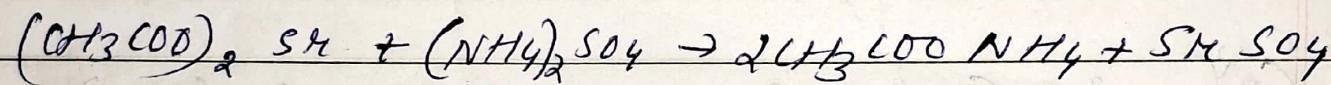
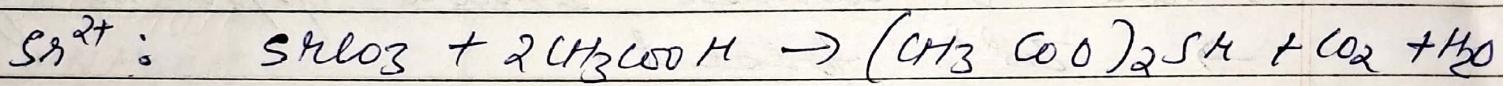
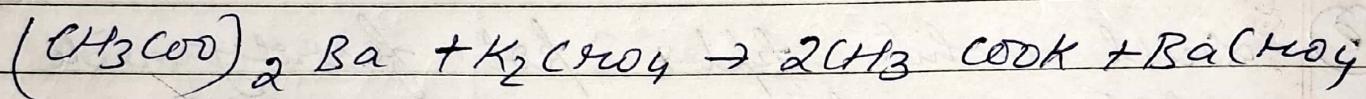
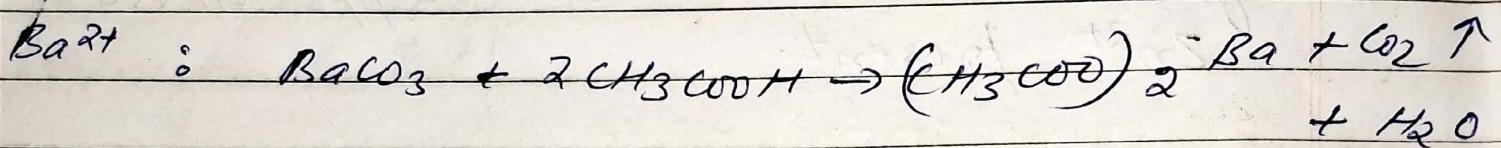


### Analysis of group V)

white ppt + dissolves in the ppt in hot dil  $\text{CH}_3\text{COOH}$  + divide the solution into three parts.

First part + potassium chromate  $\rightarrow$  yellow ppt  $\Rightarrow$   
 $Ba^{2+}$  confirmed.

Second part +  $(NH_4)_2SO_4 \rightarrow$  white ppt  $\rightarrow Sr^{2+}$  present  
Third part + Ammonium malate + Ammonium hydroxide  $\rightarrow$  white ppt  $\rightarrow Ca^{2+}$  present.



### Analysis of group VI

Sample +  $NH_4Cl + NH_4OH +$  ammonium phosphate sol $\rightarrow$  rub the sides of the test tube with a glass rod  $\rightarrow$  white ppt  $\rightarrow Mg^{2+}$  present.

### Reaction:

