PRACTICALS

Inorganic Qualitative Analysis

Instructions to the students:

- The students are required to have an observation and a record note book
- The students are advised to use overcoat and safety glass in laboratory.
- They are not permitted to taste or touch any reagent. If any reagent falls on skin, it must be immediately washed with water.
- The students should not inhale any gas or vapour directly.
- To transfer any solutions use droppers and for salts use spatula. During heating of a test tube, the open end should not face any student.
- For any accident in lab, immediately report to the teacher - incharge.
- Follow the systematic procedure carefully during analysis.
- Try to understand the chemistry in each test clearly. In the inference column there may be wording such as "Presence of or May be". Presence of means it is confirmed either the cation or anion, and May be means doubtful, further analysis is required.

List of salts

1. Lead Nitrate
2. Copper Sulphate
3. Copper Carbonate
4. Ferric Chloride
5. Zinc Sulphate
6. Zinc Sulphide
7. Aluminium Sulphate
8. Aluminium Nitrate
9. Calcium Carbonate
10. Barium Chloride
11. Ammonium Chloride
12. Ammonium Bromide
13. Magnesium Sulphate
14. Magnesium Carbonate
15. Magnesium Phosphate

Systematic analysis of a simple salt

Analysis of anions

Salt NO:

Date:

Serial NO:	Experiment	Observation	Inference
1	Note the colour of the salt	Blue/Green Brown	May be copper sulphate May be an iron salt
2	Action of heat: A small amount of a salt is strongly heated in a test tube	A colourless gas with the pungent small turning red litmus paper into blue evolves. It gives a dense white fumes when a glass rod dipped in Conc. HCl is brought close to its mouth	Presence of an ammonium salt
		A reddish brown gas with a fishy odour evolves	Presence of a nitrate salt
		Salt is yellow when hot and white when cold	May be a zinc salt
3.	Flame test: Take a small amount of salt in a watch glass. Add a drop of Conc. HCl to it and form a paste. Take the paste at the charred end of the splinter and introduce it near the Bunsen flame	Bluish green flame	Presence of a copper salt
		Apple green	Presence of a barium salt
		Brick red	Presence of a calcium salt
4.	Action of dil. HCl: Take a small amount of salt in a test tube and add about 1mL of dil. HCl to it. Gently heat it in the Bunsen flame	A colourless, odourless gas turning lime water milky evolves	Presence of carbonate
		A reddish brown gas with the fishy odour turning a moist ferrous sulphate paper brown evolves	Presence of nitrate
		A colourless gas with a rotten egg smell turning a paper dipped in lead acetate shining black evolves	Presence of sulphide

5	Action of Conc.H_2SO_4: Take a small amount of a salt in a dry test tube, add about 0.5mL of Conc. H_2SO_4 and gently heat it in the Bunsen flame	A colourless gas evolves. It gives a dense white fumes when a glass rod dipped in liquid ammonia is brought close to its mouth	Presence of chloride
		A reddish brown gas turning moist fluorescein paper green evolves	Presence of bromide
		Reddish brown gas turning acidified ferrous sulphate paper green evolves.	Presence of nitrate
6	Action of MnO_2 and Conc. H_2SO_4: Take a small amount of salt in a test tube, add pinch of MnO_2 and about 0.5mL of Conc. H_2SO_4 and gently heat it in the Bunsen flame	A greenish yellow gas turning starch iodide paper blue evolves	Presence of chloride
		A reddish brown gas turning moist fluorescein paper red evolves	Presence of bromide
7.	Action of Conc. H_2SO_4 and copper turning: Take a small quantity of salt in a dry test tube and add few copper turnings and about 1mL of Conc. H_2SO_4. Gently heat it	A reddish brown gas with fishy odour turning a moist ferrous sulphate paper brown evolves	Presence of nitrate.
8.	Action of dil. NaOH solution: To a small quantity of a salt add about 1mL of dil. NaOH solution and gently heat it.	A colourless gas with the pungent smell giving dense white fumes with a glass rod dipped in dil. HCl evolves	Presence of ammonium salt
9.	Chromyl chloride test: Take a small quantity of salt in a test tube, add a pinch of potassium dichromate and three drops Conc. H_2SO_4. Gently heat it. Pass the vapours to enter another test tube containing about 0.5mL of sodium hydroxide. If a yellow solution is obtained, add about 1mL each of dil. acetic acid and lead acetate	A yellow ppt is obtained	Presence of chloride.

Analysis with sodium carbonate extract

Preparation of sodium carbonate extract:

Take 1g of the given salt and 3g of solid sodium carbonate in a100mL beaker. Add 20g of distilled water to it. Heat the beaker with its contents on a hot plate or Bunsen burner. After boiling the solution for few mins, filter it through a filter paper in a funnel and collect the filtrate in an another beaker. The filtrate is called sodium carbonate extract.

10.	Test for halides: To about one mL of the sodium carbonate extract add dil. HNO_3 in drops with shaking until the effervescence ceases, and then add about 1mL of $AgNO_3$, and shake it well.	A curdy white precipitate(ppt) insoluble in about 1mL of dil. ammonia is formed	Presence of chloride
		A pale yellow ppt sparingly soluble in ammonia is formed	Presence oh bromide
		A black ppt is formed	Presence of sulphide
11	Test with barium chloride: To about one mL of the sodium carbonate extract, add dil. acetic acid in drops with shaking until the effervescence ceases, then add 1mL of barium chloride solution and shake it.	A white ppt is formed insoluble in dil H_2SO_4	Presence of sulphate
12	Test with lead acetate: To about 1mL of the sodium carbonate extract, add 1mL of dil acetic acid and heat it , until the effervescence ceases, and then add 1mL of lead acetate	A whit ppt soluble in excess of ammonium acetate is formed	Presence of sulphate
13	Brown ring test: To about 1mL o f the sodium carbonate extract add dil. H_2SO_4 in drops with shaking until the effervescence ceases and about 0.5mL of freshly prepared ferrous sulphate solution. Then keeping the test tube in a slanting position add Conc. H_2SO_4 along the sides of the test tube.	A brown ring is formed	Presence of nitrate

14	Ammonium molybdate test: To one portion of the extract , add dil HNO_3 until the effervescence ceases, then add about 1mL each of ammonium molybdate and Conc. HNO_3	A canary yellow ppt is formed.	Presence of phosphate
15	Test with sodium nitro bruside: To about 1mL of the sodium carbonate extract add 1mL of dil .aommonia. Then add about few drops of sodium nitro bruside.	A purple or violet colouration appears	Presence of sulphide.

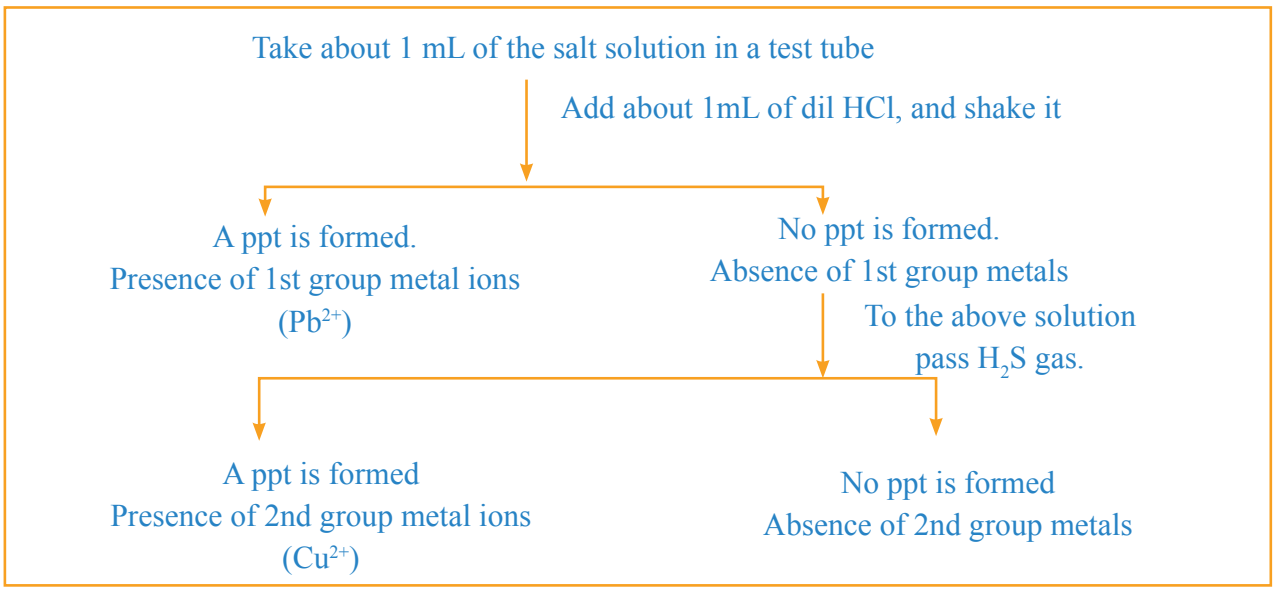
Reasoning

1	Action of heat: The reddish brown gas is NO_2 and N_2O_4 The zinc sulphate salts are yellow when hot and white when cold due to formation of semiconducting ZnO. At high temp there is bandgap excitation of electrons. When the excited electrons fall back from valence band to conduction band, light is emitted.
2	Flame test: To convert metallic salts into chlorides Conc.HCl is used. Generally chloride salts are more volatile than sulphate salts, so sulphuric acid is not used. When the metallic chlorides are introduced into flame, they are vapourised. In the middle of the flame, Cl- reduces metal ions into metal atoms. The metal atoms are then excited by the flame photons, and when the excited electrons fall back, they emit light of characteristic colour.
3	Copper turning test: When copper turning dissolves in sulphuric acid, electrons and copper ions are formed. The electrons are then used to reduce nitrate in the presence of H+ to NO2
4	Chromyl chloride test: When a chloride salt is heated in the presence of sulphuric acid, CrO_2Cl_2 chromyl chloride is formed. With NaOH it forms Na_2CrO_4 .With lead acetate it forms yellow $PbCrO_4$ ppt
5	Test for halides: If any halide ion is present in sodium carbonate extract, it reacts with silver nitrate to form , to form the silver halide ppt. chloride forms AgCl ppt, bromide forms AgBr ppt,and iodide forms AgI ppt. AgCl dissolves in ammonia by forming Ag $(NH_3)_2^+$ complex. AgBr forms little amount of complex

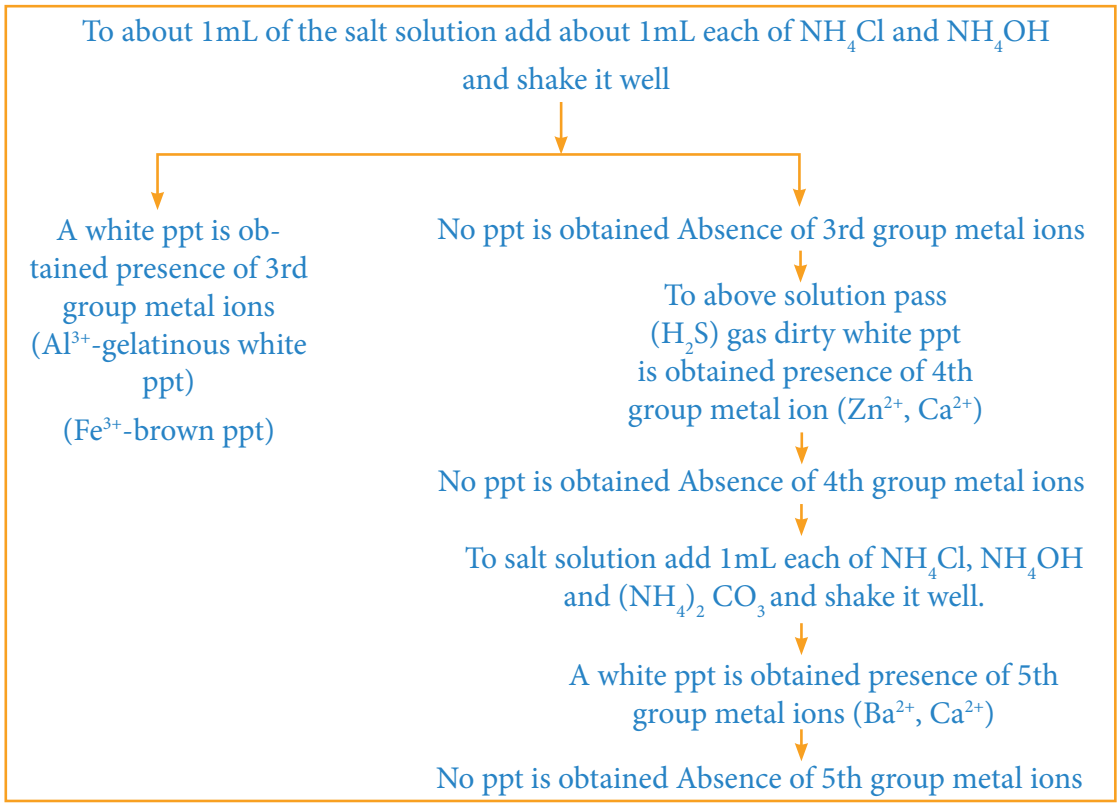
6	Test with barium chloride: Barium forms insoluble ppt with oxalate(BaC_2O_4) and sulphate($BaSO_4$) BaC_2O_4 dissolves in dil. H_2SO_4 and decolourises $KMnO_4$ when gently heated. $BaSO_4$ is insoluble.
7	Test with lead acetate: Lead acetae forms lead sulphate ($PbSO_4$) ppt
8	Brown ring test: The brown ring is due to nitroso ferrous sulphate $[Fe(NO)]SO_4$
9	Neutral ferric chloride test: The reddish brown ppt is due to $[Fe(OH)_2(CH_3COO)]$
10	Ammonium molybdate test: The canary yellow ppt is due to formation of ammonium phospho molybdate $(NH_4)_3[P(Mo_3O_{10})_4]$
11	Sodium nitro bruside test: The purple colour is due to the complex $Na_4[Fe(CN_5)NOS)]$

Preparation of solution of the simple salt for the analysis of cations:

To a small amount of salt in a test tube add 2 to 3mL of water, shake it and gently heat it. If a clear solution is obtained, directly use it for the analysis of cations. If the salt is insoluble, take a small amount of salt in an another test tube, add 3mL of dil. HCl or dil. HNO_3, shake it and gently heat it. If the salt dissolves, use the clear solution for the analysis of cations. This solution is called "original solution".



The 3rd group metal ions form metal hydroxide ppt. The 4th group metal ions form metal sulphide ppt.



Analysis of 6 group metal ions
To about 1mL of the original salt solution add about 1mL each of NH_4Cl, NH_4OH and $NH_4H_2PO_4$, and scratch the sides of the test tube. A white ppt is obtained. Presence of magnesium
To about 1mL of the original salt solution add dil. NaOH in drops with shaking. A white ppt insoluble in excess of NaOH is formed. Presence of Magnesium
To about 1mL of the original salt solution add about 1mL of Magneson reagent. A blue ppt is formed. Presence of magnesium.
To about 1mL of the original salt solution add about 1mL each of Nessler's reagent and NaOH. A chocolate brown ppt is obtained. Presence of ammonium

Reasoning:

Magnesium forms $MgNH_4PO_4$ ppt.

Maneson reagent is p-nitro azobenzene resorcinol. The blue ppt is due to precipitation of magneson by $Mg(OH)_3$

Nessler's reagent is prepared by slowly adding potassium iodide to mercury chloride. Initially a white ppt of HgI_2 is obtained. The ppt dissolves in excess forming a clear solution. This clear solution is called the "Nessler's reagent". It is $K_2[HgI_4]$

The bown ppt is due to $HgO.Hg(NH_2)I$. It is a basic mergury (ii) amido amine.

Analysis of group ppt:

Analysis of the 1st group ppt:

Experiment	Observation	Inference
To the ppt add about 1mL of water and boil it	The ppt dissolves	Presence of lead
Test for Lead: i.) To one portion of the hot solution add about 1mL of K_2CrO_4 ii.) To an another portion of the hot solution add about 1mL of KI. To the yellow ppt add about 1mL of water, boil and cool.	A yellow ppt is obtained A yellow ppt is obtained. The yellow ppt dissolves on boiling , and on cooling golden spangles appear	

Reasoning:

Lead forms $PbCrO_4$ and PbI_2 ppt - Recrystallisation of lead iodide crystals appeared as golden yellow spangles.

Analysis of the 2nd group ppt:

To the ppt add about 1mL of dil HNO_3 and boil it.The ppt dissolves. Cool it.

i). To one portion of the solution add ammonium hydroxide	No ppt is obtained, but the solution is blue	Presence of copper
iii) Test for copper:To the blue coloured solution add about 1mL each of acetic acid and potassium ferrocyanide	A red brown ppt is obtained	Presence of copper

Reasoning

Prepare sodium stannite solution by mixing equal volume of about 1mL each of stannous chloride and sodium hydroxide.

With NH_4OH copper forms soluble $[Cu(NH_3)_4]^{2+}$ complex,

Copper forms a brown ppt of $K_2Cu[Fe(CN)_6]$

Analysis of the 3rd group ppt:

To the ppt add a pinch of sodium peroxide and boil it	A red or brown ppt is obtained A colourless solution is obtained	Presence of iron Presence of aluminium
i.) Test for iron:To one portion of the red ppt add about 1mL of dil HCl and boil it and then add about 1mL of potassium ferocyanide	A blue ppt is obtained	Presence of iron
ii.) To an another portion of the ppt add about 1mL of dil. HNO_3 boil it and then add about 1ml of KCNS	A blood red colouration is seen	Presence of iron
iii.) Test for aluminium:To the colourless solution add dil.HCl and shake it	A gelatinous white ppt is obtained	Presence of aluminium

Reasoning:
Obtain sodium peroxide by mixing equal volume of about 1mL each of NaOH and H_2O_2
Iron forms a blue ppt(prussian blue) of $Fe_4[Fe(CN)_6]_3$
Iron forms $[Fe(CN)_6]^{3-}$ complex which is blood red coloured.
Aluminium forms a gelatinous white ppt of $Al(OH)_3$

Analysis of the 4th group ppt

To the ppt add dil HCl and boil it	The ppt dissolves	Presence of zinc
i.)Test for zinc To the solution add about 1.5mL of dil. NaOH and boil it	A clear solution is obtained	Presence of zinc

Reasoning:
Zinc initially forms $Zn(OH)_2$ ppt, and it dissolves in excess to form sodium zincate (Na_2ZnO_2)
Zinc forms white ppt of ZnS

Analysis of the 5th group ppt:

To the ppt add about 1mL of dil. acetic acid and gently heat it. The ppt dissolves. Divide the solution into two portions. i).To one portion add about 1mL of potassium chromate	A yellow ppt is obtained. Filter the ppt using a funnel and filter paper, and transfer the residue to a watch glass. Add a drop of Conc. HCl. Take a portion of the paste at the charred end of a splinter and introduce near the Bunsen flame. A transient green is imparted to the flame	Presence of barium.
ii). To an another portion add about 1mL of ammonium sulphate	A white ppt is obtained. Filter the ppt using a filter paper and funnel. Transfer the residue to a watch glass. Add a drop of Conc. HCl. Take the residue at the charred end of the splinter and introduce near the Bunsen flame. A crimson red colour is seen If no ppt is obtained, to the solution add about 1mL of potassium ferrocyanide and shake it. A pale yellow ppt appears.	Presence of calcium

Reasoning:

Barium forms a yellow $BaCrO_4$ ppt

The pale yellow ppt of calcium is due to $Ca_2[Fe(CN)_6]$