QUALITATIVE INORGANIC ANALYSIS

Preliminary Tests

# COLOUR OF THE SOLID COMPOUND :	INFERENCE
Blue or bluish green	Cu^{2+}
Green	Ni^{2+}
Dark brown	Fe^{3+}
Light pink or flesh colour	Mn^{2+}
Rose red or violet	Co^{2+}
White	Absence of Cu^{2+}, Ni^{2+},Fe^{3+},Ca^{2+}

# ODOUR :	INFERENCE
Vinegar link smell	CH_3COO^-
Rotten eggs smell	S^{2-}
Smell of SO^2 gas	SO_3^{2-}
Ammoniacal smell	NH_4^+

# DRY HEATING TEST :	INFERENCE
Colorless & odourless gas	CO_3^{2-}
Colorless gas with rotten eggs smell	S^{2-}
White fumes with suffocating odour	SO_3^{2-}
Brown color gas	NO^{2-}, NO^{3-}, Br^-
Yellowish green gas with pungent smell	Cl^-
Violet vapour	I^-
Characteristic vinegar like smell	CH_3COO^-
White when hot and blue when cold	Cu^{2+}
Yellow when hot and white when cold	Zn^{2+}
Blue when hot and pink in cold	CO^{2+}
White residue, glows on heating	Mg^{2+},Al^{3+}
Black residue	CO^{2+}, Fe^{3+}, Ni^{2+} or Mn^{2+}

# COBALT NITRATE TEST:	Inference
Green mass	Zn^{2+}
Pink mass	Mg^{2+}
Blue mass	Al^{3+}, PO_4^{3-}, BO_3^{3-}
Dirty green	Sn^{2+}
Black	No definite indication

# FLAME TEST :		
Color of flame observed with naked eyes	Color of flame observed through blue glass	INFERENCE
Persistent golden yellow	Invisible pink	Na^+
Violet flame of shot duration	Pink	K^+
Brick red flame of short duration	Light yellow	Ca^{2+}
Apple green flame	Bluish green	Ba^{2+}
Crimson	Purple	Sr^{2+}
Intense blue surrounded by green flame	Same color visible	Cu^{2+}

# Borax Bead Test : Only for coloured salts (copper, iron, manganese, nickel, cobalt, chromium salts)				
Color of the bead in oxidizing flame		Color of the bead in reducing flames		INFERENCE
WHEN HOT	WHEN COLD	WHEN HOT	WHEN COLD	
Green	blue	Colorless	Red	Cu^{2+}
Yellow	Yellow	Green	Yellow	Fe^{3+}
Green	Green	Green	Green	Cr^{3+}
Pink	Pink	Colorless	Colorless	Mn^{2+}
Brown	Pale brown	Grey or black	Grey or black	Ni^{2+}

ANALYSIS FOR ANIONS

# DILUTE SULPHURIC ACID TEST:	INFERENCE
Effervescence with colorless & odourless gas turns lime water milk	CO_3^{2-}
Brown colored gas turning starch iodide paper blue	NO_2
Colorless gas with rotten egg smell turning lead acetate paper black or grey	S^{2-}
Colorless gas with suffocating odour turning potassium dichromate paper green	SO_3^{2-}
Colorless vapour having smell of vinegar	CH_3COO^-

# POTASSIUM PERMANGANATE TEST:	INFERENCE
Purple colour of $KMnO_4$ solution is discharged	
Yellowish green gas evolved	Cl^- (may be)
Red brown gas evolved	Br^- (may be)
Violet vapours	I^- (may be)
No gas evolved but purple colour is discharged	Fe^{2+} (may be)

# CONCENTRATED SULPHURIC ACID TEST:	INFERENCE
Colorless gas pungent small. Gives dense white fumes with rod dipped in NH_4OH	Cl^-
Brownish gas with pungent smell. Intensity of fumes increases on adding solid MnO_2	Br^-
Evolution of violet vapours turning starch paper blue	I^-
Brown fumes intensifying on adding Cu turning or bits of filter paper	NO_3^-

CONFIRMATORY TEST OF ANIONS

TEST	OBSERVATION
1.CARBONATE (CO_3^{2-}) #0.2g salts+2ml Dil.H_2SO_4 pass through lime water #1ml W.E+ equal amount of $MgSO_4$	Lime water turns milky due to formation of $CaCO_3$ White precipitates of $MgCO_3$
2.SULPHITE (SO_3^{2-}) # About 1ml of W.E.+ Acetic acid + 2ml $BaCl_2$ # 1ml of PPT. solution obtained + 2drops of $KMnO_4$ solution.	White ppt. soluble in dil.HCl Pink color of $KMnO_4$ discharges
3.SULPHIDE (S^{2-}) #1ml of W.E + a few drops of freshly prepared sod. Nitroprusside solution. #1ml W.E. + Acetic acid + 1ml lead acetate solution. # shake about 2ml of S.E. with about 0.2g of freshly prepared cadmium carbonate	Purple or violet coloration Black ppt. of PbS. Formation of yellow ppt. of CdS.
4.NITRITE (NO_2^-) # 2ml of W.E.+ dil.Acetic acid + a pinch of thiourea and few drops of ferric chloride sol. # 3-4 drop of aq. Sol.+ 2 drop of sulphanilic acid + 2 drop of napthylamine into it # 1ml of W. Extract + few drop of diphenylamine reagent solution.	A blood red coloration Pink coloration A deep blue coloration
5. ACETATE (CH_3COO^-) # 1 ml W. Extract + 1ml of neutral ferric chloride # Heat the pinch of mixture with few drops of conc.H_2SO_4 and 0.5ml of ethyl alcohol in a test tube.	A blood red coloration Pleasant fruity smell due to the formation of ethyl acetate.
6. CHLORIDE (Cl^-) # 1ml of S.E.+ dil. HNO_3+1ml of $AgNO_3$ sol. In it. # Heat about 0.2g of the solid mixture with 0.4g of MNO_2 + 2ml conc.H_2SO_4. # 0.2g of mixture + 0.3g potassium dichromate + 3ml conc. H_2SO_4 and heat pass reddish gas through NaOH sol. + acetic acid + few drop of lead acetate sol. In it.	Crudy white ppt. soluble in NH_4OH Evolution of greenish yellow gas With pungent smell. Yellow ppt. of lead chromate.
7.BROMIDE (Br^-) #2ml W.E.+dil.HNO_3+2ml of $AgNO_3$ solution. #2ml of W.E.+ Acetic acid+1-2ml of CS or $CHCl_3$ to it. Add freshly prepared Cl_2 water or dil. HNO_3 with constant shaking in the tube.	Light yellow ppt. partially soluble in NH_4OH A brown color in the organic layer.
8.IODIDE (I^-) # Neutralize 1-2 ml of the S.E. with acidic acid + few drops of silver nitrate solution. # 2ml of S.E.+ dil. Aceticacid+2ml of CS or CCl_4 drop wise with shaking Cl_2 water or HNO_3.	Pale yellow ppt. insoluble in NH_4OH A violet color in the organic layer confirms iodide.
9.NITRATE (NO_3^-) #1ml of W.E. + diphenylamine reagent solution. #2-3ml of W.E.+acetic acid+10 drops of ferrous sulphate+2ml of conc.H_2SO_4.	Blue color. Formation of brown ring at the junction of two layers.
10.SULPHATE (SO_4^{2-}) # 1ml of W.E.+dil. HCl+2ml of $BaCl_2$ solution in it. # 2ml W.E.+acetic acid +few drops of lead acetate solution in it.	White ppt. insoluble in conc.HNO_3 A white ppt. soluble in excess of hot ammonium acetate solution.
11.PHOSPHATE (PO_4^{3-}) # 2ml W.E.+dil.HNO_3+1ml of conc. HNO_3+ pinch of solid ammonium molybdate into it. # 2ml W.E.+dil HCl boil off CO_2+ few drops of magnesia mixture solution.	Yellow ppt. White crystalline ppt.

ANALYSIS FOR CATIONS

<table>
<tr><td colspan="6">To the solution of compound add dil. HCL</td></tr>
<tr><td rowspan="4">White ppt. [GroupI] Pb^{2+}</td><td>Filtrate</td><td colspan="4">Pass H_2S gas through the filtrate</td></tr>
<tr><td rowspan="3">ppt.
[Group II]
Black:
Pb^{2+}, Cu^{2+}, Hg^{2-}
Yellow:
Cd^{2-}, As^{3+}</td><td colspan="4">Filtrate. Boil off H_2S. Add 3-4 drops of conc. HNO3+add solid ammonium chloride. Boil again, cool & add ammonium hydroxide until ihe solution becomes basic.</td></tr>
<tr><td rowspan="2">ppt.
[Group III]
Reddish brown:
Fe^{3+}
White:
Al^{3+}</td><td colspan="3">Filtrate. Add solid NH_4Cl+NH_4OH+pass H_2S gas</td></tr>
<tr><td>ppt.
[Group IV]
Black:
Ni^{2+},CO^{2+}
Flesh colored:
Mn^{2+}
Dirty white
Zn^{2+}</td><td colspan="2">Filtrate. Boil off H_2S+add NH_4Cl &NH_4OH+$(NH_4)_2CO_3$ solution warm and allow to stand.</td></tr>
<tr><td>White PPT.
[Group V]
Ba^{2+},Sr^{-}
& Ca^{2+}</td><td>Filtrate.AddNH_4Cl,
NH_4OH & ammonium hydrogen phosphate.
White PPT.: Mg^{2+}
Filtrate. Heat to dryness &test for Na^{2+}& k^{+} with the residue.
Original mixture. Heat with NaOH solution.
Evolution NH_3, NH_4^{+}</td></tr>
</table>

ANALYSIS OF GROUP ZERO (NH_4^{+})

Experiment	Observation	Inference
NaOH Test	Dense white fumes	NH_4^{+} confirmed
Nessler's reagent	Reddish brown ppt.	NH_4^{+} confirmed

ANALYSIS OF GROUP I [Pb^{2+}]

Boil the white precipitate with 5.10ml of water. Divide the solution into three parts.

Confirmation

1. Cool one part under tap. White crystalline ppt. separate out.
2. Potassium iodide test to the second part, add KI solution-yellow ppt. confirms Pb^{2+}.
3. Potassium chromate test to the third part add K_2CrO_4 solution –yellow ppt. confirms Pb^{2+}.

ANALYSIS OF BLACK PRECIPITATE (PbS, CuS, HgS)

Group II cations can be put into two groups on the basis of color of their sulphates.

BLACK RESIDUE	FILTRATE	
	Bluish green	Colorless
HgS ppt. Doesn't dissolve in 50% hot nitric acid. So if black residue is left, test Hg^{2+} ions.	Test for Cu^{2+} ions. Divide the solution into two parts	Test for Pb^{2+} ions. To the sol. Add 1ml of ethyl alchohol+2ml conc. H_2SO_4. White ppt. indicates presence of Pb^{2+}. Dissolve the white ppt. in 4-5 ml of hot saturated ammonium acetate sol. & divide the solution
1. To one part adds stannous chloride. A white ppt. Turing gray confirms Hg^{2-} ion presence.	1.To one part add NH_4OH solution in excess deep blue color confirms Cu^{2+}.	1. To one part, add potassium chromate solution solution a yellow ppt. confirms Pb^{2+} ions.
2. To the other part, add a piece of clean copper wire or foil and set aside. A gray deposit, which on polishing with filter paper becomes silvery white, confirms Hg presence.	2. Acidify the second part with acetic acid +potassium ferrocynide solution chocolate color PPT. confirms Cu^{2+} presence.	2.to second part add KI solution. Formation of yellow ppt. confirms Pb^{2+} ions.

Identification of Cd^{2+} & As^{3+} [yellow ppt.] add about 3-4 ml of yellow ammonium sulphide to ppt. and warm the test tubes for 3 minutes.

CADMIUM [Cd^{2+}]	ARSENIC [As^{3+}]
Reside indicates the presence of Cd^{2+} dissolve the ppt. In dill. HCl and divide solution into two parts.	Dissolution of the ppt. Indicates presence of As^{3+}. Add dil. HCl to above solution. Appearance of yellow ppt. in hot conc. HNO_3 and divide the solution in two parts.
i) Dilute one part about 5 times with distilled water+pass H_2S gas through the resultant sol. Yellow ppt. confirms Cd^{2+}.	i) To one part+ammonium molybdate sol. Formation of yellow ppt. confirms As^{3+}.
ii) To the second part add sodium hydroxide sol. Formation of white ppt. Confirms Cd^{2+}.	ii) To the second part add ammonium hydroxide + magnesia mixture. Appearance of white ppt. confirms As^{3+}.

ANALYSIS OF GROUP III [CATIONS]	
Reddish brown pppt. –Fe^{3+} present	**White gelatin ppt. –Al^{3+} present**
Dissolve the ppt. In minimum amount of HCl and dissolve the sol. In two parts	Dissolve the ppt. By boiling with NaOH.divide the solution in two parts
i) To one part add potassium ferrocynide solution. A blue color confirms Fe^{3+}.	i) To one part add NH_4Cl and boil. Formation of white gelatin ppt.confirms Al^{3+}.
ii) To the second part add potassium sulphocynide solution. Appearance of deep red coloration confirms Fe^{3+}.	ii) Take second part, acidify with dil. HCl. Add two drops of blue litmus followed by ammonia till it smell ammonia. Blue ppt. Floating in colorless solution confirms Al^{3+}.
Distinction between Fe^{2+} and Fe^{3+}. Add a few drops of potassium ferricyanide solution to the O.S. before adding the conc. HNO_3.	
Appearance of deep blue colour or ppt. indicates the presence of Fe^{2+}.	
Appearance of a deep blue coloration or ppt. On adding potassium ferrocyanide sol. indicates the presence of Fe^{3+} to the original solution.	

ANALYSIS OF GROUP IV [CATIONS]		
BLACK RESIDUE	**Pink / White ppt. Dis. ppt in HCl**	
Dissolve the minimum quantity of aqua regia. Evaporate the solution to almost dryness in a porcelain dish. Extract the residue with 5ml of distilled water. Change in color from blue to pink confirms CO^{2+}. Divide the extract in two parts.	Boil to expel H_2S gas. Cool it and add excess of NaOH solution into it.	
i) To one part add 1ml of ethyl alcohol+100gm of NH_4SCN.shake well. Blue color in alcohol layer confirms the presence of CO^{2+} in the mixture.	Dissolve the residue in conc. HNO_3+ 0.2g PbO and boil. Purple solution confirms Mn^{2+}.	Pass H_2S gas through it. White ppt. Confirms Zn^{2+} presence in the mixture.
ii) To the second part add 5ml of dimethyl glyxoime and NH_4OH. Pink ppt. confirms the presence of Ni^{2+} in the mixture.		

ANALYSIS OF GROUP V [CATIONS]		
RESIDUE	**FILTRATE**	
Residue $BaCrO_4$, wash the ppt. with hot water and perform the flam test. Apple green flame confirms the presence of Ba^{2+}.	Filter may contain Sr^{2+} & Ca^{2+}. Add excess of ammonium sulphate solution & heat. Allow it to stand & filter.	
	Residue white $SrSO_4$ confirms by flame test. Persistent crimson red flame indicates Sr^{2+}.	Filtrate. Add ammonium oxalate solution & warm. White ppt. dissolves in HCl & confirms by flame test. Brick red flame Ca^{2+} confirmed.

IDENTIFICATION OF Mg2+
Divide the filtrate of Group V in two parts.
To one part add 1-2ml of disodium hydrogen phosphate solution followed by ammonia solution till mixture smells ammonia – White crystalline precipitate . To the second part add magneson reagent (an alkaline solution of p-nitrobenzene azo resorcinol) and excess of NaOH solution – Sky blue precipitate.

Some Hints for Analysing a Salt :

- ***If the salt is white in colour ,then Copper ,iron, cobalt , nickel cations are not present.***
- ***If the salt contains sulphate as acid radical (anion)then Ba^{2+},Sr^{2+},$Ca^{2+}$and Pb^{2+} should be absent. (Sulphates of these cations are insoluble & insoluble salts are not in the syllabus.***
- ***When Phosphate is confirmed as anion , radicals of Group IV & V & Magnesium should be absent.***
- ***To ensure the presence of Group V , take about 1 ml of Group II filtrate & add about 1 ml alcohol & then slowly add 1ml of H_2SO_4.***
  Appearance of white precipitates indicates the presence of Group V.

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