CEMA CC6-13P Lab Course Instructions
Qualitative Analysis of an Inorganic Sample

1. Preliminary Investigation

Colour –
Nature – crystalline/amorphous
Solubility – water (cold/hot); dil. HCl (cold/hot); aqua regia ; [Else insoluble]

2. Dry Tests for Basic Radicals

Experiment	Observation	Inference
1.Sample heated in a dry test tube.	(i) yellow/brown when hot, yellow when cold (ii)yellow when hot, white when cold (iii) brown/black when hot, brown when cold (iv) black residue (v) green residue (vi) water drops condense in upper cooler part of test tube (vii) greenish yellow gas, pungent smell, turns starch iodide paper blue (viii) violet vapours sublime as black crystals (ix)brown gas (x)H_2S gas smell of rotten eggs, turns lead acetate paper black (xi) white sublimate, turns yellow with H_2S (xii)white sublimate, no colour change with H_2S, smell of ammonia (xiii) the substance swells on heating	(i)Pb salts, SnO_2 (ii) Zn salts (iii) Fe salts (iv) Mn, Co, Ni, Cu salts (v)Cr salts (vi) hydrated salts (vii)chloride salts (viii) iodide salts (ix)nitrate, nitrite or bromide salts (x) sulphide salts (xi) As_2O_3 (xii) NH_4^+ salts (xiii) borax
1A. The white sublimate heated strongly with NaOH and CaO (soda lime)	(i)smell of ammonia, Nessler's reagent turns brown (ii)shiny black mirror like appearance inside the test tube	(i)NH_4^+ salts (ii)As salts
2. Flame test, with clean platinum wire, conc. HCl and sample.	(i)golden yellow, colourless through blue glass (ii)lilac (pale violet) (iii)transient brick red (deep red) (iv)persistent crimson (v)apple green (vi)bluish green (vii)green, lambent green (viii) lambent blue	(i)Na salts (ii)K salts (iii) Ca salts (CaF_2) (iv) Sr salts (v) Ba salts (vi)Cu salts (vii)BO_3^{3-}, Pb, As salts (viii)Sn salts

3.Borax bead test. A transparent bead in a Pt wire loop prepared with borax. 2-3 crystals of sample fused with the bead in a Bunsen flame.	(i)green when hot, blue when cold, (sometimes red) (ii) dark blue (iii)green (iv)reddish yellow when hot, yellow when old (v)violet (amethyst colour) (vi) reddish brown **[performed for coloured salts only]**	(i)Cu salts (ii)Co salts (iii)Cr salts (iv)Fe salts (v)Mn salts (vi)Ni salts
4.Oxidative Fusion test. Sample heated on a mica foil with Na_2CO_3, NaOH and KNO_3, till the mixture fuses.	(i)yellow melt; Aqueous extract forms yellow precipitate with acetic acid and lead acetate solution. (ii)green melt; Aqueous extract gives pink coloration with acetic acid.	(i)Cr salts (ii)Mn salts
5.Fluorescence test for Sn. Sample, with 1:1 HCl and Zn granules taken in a beaker, is stirred with a test tube half-filled with water, and the test tube held over a Bunsen flame.	Blue fluorescence observed in the outer wall and the bottom of the test tube	SnO_2, Sn salts
6.Test for ammonium. Sample heated with aqueous NaOH.	Pungent smell of ammonia	NH_4^+ salts
7.Tests with HCl extracts. Sample dissolved in HCl, by heating if necessary, and solution divided into 7 parts.	(i)yellow precipitate with KI solution (ii)white precipitate with dil. H_2SO_4 (iii)deep blue coloration with $K_4[Fe(CN)_6]$ (iv) blood red coloration with $NH_4(SCN)$ (v)yellow coloration o heating with NaOH and H_2O_2 (vi)blue alcoholic layer on adding amyl alcohol and $NH_4(SCN)$ (vii)white precipitate on boiling with NaOH and passing H_2S	(i)Pb salts (ii)Pb, Ca, Sr, Ba salts (iii)Fe salts (iv)Fe salts (v)Cr salts (vi)Co salts (vii) Zn salts
8. Tests with HNO_3 (1:1) extracts. Sample dissolved in HNO_3, by heating if necessary, and the	(i)pink coloration with solid $NaBiO_3$ (ii)red precipitate with excess ammonia and DMG solution (iii)deep blue coloration with ammonia, then chocolate brown precipitate with acetic acid and $K_4[Fe(CN)_6]$	(i)Mn salts (ii)Ni salts (iii)Cu salts

solution divided into 3 parts.		
9.Tests with NaOH extracts. Sample dissolved in NaOH by boiling, and extract divided into 3 parts.	(i)white gelatinous precipitate on adding excess solid NH_4Cl (ii)White precipitate on adding acetic acid and (a) passing H_2S (b) adding $K_4[Fe(CN)_6$ soln. White precipitate dissolves in NaOH (iii) yellow precipitate on adding acetic acid and K_2CrO_4 solution	(i)Al salts (ii)Zn salts (iii)Pb salts
10. Sample heated with Na_2CO_3 and a few drops of water.	Pungent smell of ammonia, producing dense white fumes with a glass rod dipped in dil HCl.	NH_4^+ salts

3. Dry Tests for Acid Radicals

Experiment	Observation	Inference
11.Sample heated with dil. H_2SO_4 [Test repeated with addition of Zn dust]	(i)colourless gas with rotten egg smell, turns lead acetate paper shiny black (ii)brown fumes, turns starch-KI paper blue-black (iii) colourless gas with smell of burnt sulphur which turns acidified $K_2Cr_2O_7$ paper green, along with deposition of sulphur (turbidity) [colourless gas with rotten egg smell, turns lead acetate paper black]	(i)S^{2-} salts (ii)NO_2^- salts (iii) $S_2O_3^{2-}$ salts [covalent sulphides]
12.Sample heated with conc. H_2SO_4	(i)colourless gas, forms dense white fumes with ammonia (ii) colourless gas, turns water drop milky /waxy, test tube inner wall appears oily (iii)brown fumes, intensifies on adding Cu-turnings (iv)reddish brown vapours (v)violet vapours	(i)Cl^- salts (ii)F^- salts (iii) NO_3^- salts (iv)Br^-, BrO_3^- salts (v)I^- salts
13.Sample heated with conc. H_2SO_4 and MnO_2	(i)colourless gas with smell like bleaching powder, turns starch-KI paper blue. (ii)reddish orange vapours (iii)violet vapours	(i)Cl^- salts (ii)Br^- salts (iii)I^- salts

14.Sample warmed with Zn granules and dil. H_2SO_4. The evolved gas passed into NaOH solution, and $Na_2[Fe(CN)_5NO]$ solution added.	Colourless gas evolves with smell of rotten eggs, which turns lead acetate paper black. Violet coloration	S^{2-} salts
15.Sample heated with NaOH and Zn dust.	Evolution of ammonia	NO_3^-, NO_2^- salts
16.Sample heated with KI and dil. H_2SO_4.	Violet vapours evolved	NO_2^-, IO_3^- salts
17.Sample heated with KBr and dil. H_2SO_4.	Reddish brown vapours evoled	BrO_3^- salts
18.Sample heated with conc. HNO_3 and ammonium molybdate solution. A.Yellow precipitate boiled with ammonium acetate solution and then cooled. B. Sample again boiled with conc. HNO_3, ammonium molybdate solution and tartaric acid.	Canary yellow precipitate (i)Precipitate dissolves (ii) Precipitate dissolves, then forms a white precipitate on cooling (i)no precipitate (ii)yellow precipitate	PO_4^{3-} , AsO_4^{3-} salts (i) PO_4^{3-} salts (ii) AsO_4^{3-} salts (i) AsO_4^{3-} salts (ii) PO_4^{3-} salts
19.Solid sample, methyl or ethyl alcohol and conc. H_2SO_4 mixed and the evolved vapours ignited in the Bunsen flame.	The vapours burn with a green flame at the mouth of the test tube	BO_3^{3-} salts
20.Solid sample added to Iodine – sodium azide solution on a watch glass.	Decolourisation of iodine and evolution of N_2 gas (bubbles)	S^{2-}, $S_2O_3^{2-}$, SCN^- salts

4. Wet Tests for Acid Radicals

A. Confirmatory Tests

The sample is dissolved in 15ml distilled water **OR** boiled with 2-3 times its weight of Na_2CO_3 and 15ml water. It is filtered (sodium carbonate extract). With the **Aqueous solution OR Sodium carbonate extract** the following tests are performed.

Experiment	Observation	Inference
1.To the solution sodium nitroprusside soln is added.	Violet coloration	S^{2-} salts
2.To the solution dil. HCl and $BaNO_3$ / $BaCl_2$ soln is added.	Heavy white precipitate insoluble in dil. HCl or HNO_3	SO_4^{2-} salts

3.To the neutral solution $FeCl_3$ soln is added.	(i) blood red colouration (ii) Prussian blue colouration	(i)SCN^- salts (ii)$[Fe(CN)_6]^{4-}$ salts
4. To the neutral solution, freshly prepared $FeSO_4$ soln is added.	Blue coloration	$[Fe(CN)_6]^{4-}$ salts
5.To the solution freshly prepared $FeSO_4$ solution is added, followed by conc. H_2SO_4 carefully down the sides of the test tube.	(i)Brown ring formed at the junction of the two liquids (ii)whole solution turns brown (even if test is performed with dil. H_2SO_4)	(ii)NO_3^- salts (ii)NO_2^- salts
6.To a pinch of the solid sample in a watch glass, sulphanilic acid and α-naphthylamine solution is added.	(i)red coloration (ii)red coloration after adding Zn dust	(i) NO_2^- salts (ii) NO_3^- salts
7.To the solution dil. HNO_3 and $AgNO_3$ soln is added.	(i)curdy white precipitate insoluble in HNO_3 but soluble in NH_4OH (ii)pale yellow precipitate partially soluble in NH_4OH (iii)yellow precipitate insoluble in NH_4OH	(i)Cl^-, SCN^- salts (ii)Br^- salts (iii)I^- salts
8.Solid sample, solid $K_2Cr_2O_7$ and conc. H_2SO_4 is heated in a test tube, the evolved vapours passed into NaOH solution. The yellow solution is acidified with acetic acid and lead acetate solution added.	Intense reddish brown fumes evolve, which turns NaOH solution yellow Yellow precipitate obtained	CrO_2Cl_2 vapours Cl^- salts
9.A paste is made with the solid sample, CaF_2 and conc. H_2SO_4 in a watch glass. With a glass rod, a little of the paste is held close to the Bunsen flame.	Green flame coloration	BO_3^{3-} salts
10.A paste is made with the solid sample, borax and conc. H_2SO_4 in a watch glass. With a glass rod, a little of the paste is held close to the Bunsen flame. To the sample solution in HCl, Zirconyl nitrate solution	Green flame coloration Pink colour of the solution turns yellow	F^- salts

and Alizarin-S solution is added. To the sample solution in HCl, $FeCl_3$ solution and NH_4SCN solution is added.	Blood red colour of the solution discharged	
11.To the neutral solution $CaCl_2$ solution is added.	(i)white precipitate insoluble in acetic acid (ii)white precipitate soluble in acetic acid	(i)F^- salts (ii)PO_4^{3-}, AsO_4^{3-} salts
12.To the solution dil. HCl, $CHCl_3$ and Chlorine water is added and shaken	(i)organic layer turns brown (ii)organic layer turns violet	(i)Br^- salts (ii)I^- salts
13. To the neutral solution saturated $MnSO_4$ solution is added.	A transient red colour observed. On heating it turns brown, and brown precipitate dissolves in dil. H_2SO_4 and oxalic acid.	BrO_3- salts
14. To the solution SO_2-water is added, boiled, cooled and $AgNO_3$ solution added.	Yellow precipitate	IO_3^- salts
15.If solution is yellow it is acidified with dil. acetic acid. To the solution lead acetate soln is added.	Solution turns orange Yellow precipitate	CrO_4^{2-} salts $CrO_4^{2-}/Cr_2O_7^{2-}$ salts

B. Unambiguous detection of Allied Radicals.

Experiment	**Observation**	**Inference**
Nitrate and Nitrite To the aqueous solution or Na_2CO_3 extract one drop each of sulphanilic acid and α-naphthylamine is added.	Red colouration	NO_2^- confirmed
The aqueous solution or Na_2CO_3 extract is boiled with urea and dil H_2SO_4, cooled, Zn dust and one drop each of sulphanilic acid and α-naphthylamine is added.	Red colouration	NO_3^- confirmed

Chloride, Bromide and Iodide To the aqueous solution or Na_2CO_3 extract, dil HCl, CCl_4 or $CHCl_3$ and Cl_2-water is added and shaken. More Cl_2-water is added and shaken. To the aqueous solution or Na_2CO_3 extract, acetic acid and PbO_2 is added and boiled to expel Br_2 and I_2. It is filtered, dil HNO_3 and $AgNO_3$ solution added.	Violet colouration of organic layer Violet colour disappears and organic layer turns brown Curdy white precipitate soluble in NH_4OH	I confirmed Br^- confirmed Cl^- confirmed
Sulphide, Sulphate and Thiosulphate The aqueous solution or Na_2CO_3 extract is shaken with excess $CdCO_3$ and filtered. Filtrate shaken with excess $Sr(NO_3)_2$ solution and filtered. Filtrate acidified with dil HCl and boiled. OR To the filtrate $AgNO_3$ solution added.	Yellow precipitate White precipitate insoluble in c. HCl SO_2 gas evolved and solution becomes turbid. White precipitate turns brown then black	S^{2-} confirmed SO_4^{2-} confirmed $S_2O_3^{2-}$ confirmed
Phosphate and Arsenate The aqueous solution or Na_2CO_3 extract is shaken with $Mg(NO_3)_2$ solution and filtered. The white precipitate dissolved in HCl and H_2S passed and filtered. H_2S boiled off, ammonium molybdate solution added and boiled again.	White precipitate Yellow precipitate Canary yellow precipitate	PO_4^{3-} and AsO_4^{3-} AsO_4^{3-} confirmed PO_4^{3-} confirmed

Ferrocyanide, Ferricyanide and Thiocyanate The aqueous solution or Na_2CO_3 extract is boiled with dil HCl, divided into two parts. To one part $FeCl_3$ solution and amyl alcohol is added. To the other part dil H_2SO_4 and $FeSO_4$ solution is added.	Organic layer becomes red Aqueous layer becomes blue. Deep blue colouration	SCN^- confirmed $[Fe(CN)_6]^{4-}$ confirmed $[Fe(CN)_6]^{3-}$ confirmed
Bromide and Bromate To the aqueous solution or Na_2CO_3 extract $AgNO_3$ solution is added and filtered. Filtrate boiled with SO_2-water.	Pale yellow precipitate soluble in conc. NH_4OH Pale yellow precipitate soluble in conc. NH_4OH	Br^- confirmed BrO_3^- confirmed
Iodide and Iodate To the neutral Na_2CO_3 extract CCl_4 and Cl_2 water is added. To the neutral Na_2CO_3 extract excess $AgSO_4$ solution is added, AgI filtered off, filtrate boiled with SO_2, then dil. HNO_3 and $AgNO_3$ solution is added.	Organic layer turns violet Yellow precipitate	confirmed IO_3^- confirmed

5. **Wet Test of Basic Radicals**

A. **Systematic Group Separation:** The sample is dissolved in distilled water **OR** dil. HCl **OR** aqua regia. With the **Aqueous solution OR HCl extract** the following tests are performed. [Any residue after obtaining the aqueous and acid extracts is analysed for **insoluble compounds**]

To the sample solution dil HCl is added and the precipitate filtered.	
Group I White ($PbCl_2$)	*[A few drops of H_2O_2 added to the filtrate to oxidise Sn^{2+} to Sn^{4+}.] The filtrate is warmed, dil HCl is added and H_2S is passed through. The precipitate is filtered.

<table>
<tr><td rowspan="4"></td><td rowspan="4">Group IIA
Black
(CuS, PbS, Bi_2S_3)
Yellow
(CdS)

Group IIB
Yellow
(As_2S_3, SnS, SnS_2)
Orange
(Sb_2S_3)</td><td colspan="3">H_2S is boiled off. *[A few drops conc. HNO_3 is added and boiled to oxidise Fe^{2+} to Fe^{3+}.]
*The solution is evaporated to dryness 2-3 times with conc. HCl to remove BO_3^{3-} and F^-.
** To the solution glacial acetic acid and lead acetate soln is added, the white precipitate (PO_4^{3-}) filtered off. Dil HCl is added and H_2S is passed, black PbS filtered off.
To the filtrate excess solid NH_4Cl and NH_4OH is added and the precipitate filtered.</td></tr>
<tr><td rowspan="3">Group IIIA
Brown
$Fe(OH)_3$,
$MnO_2.H_2O$
White($Al(OH)_3$)
Green($Cr(OH)_3$)</td><td colspan="2">To the filtrate NH_4OH is added, heated to boiling. H_2S is passed, and the precipitate filtered.</td></tr>
<tr><td rowspan="2">Group IIIB
Black
(NiS, CoS)
Pink (MnS)
White (ZnS)</td><td colspan="2">Filtrate evaporated to ¼ its volume, NH_4OH, NH_4Cl, and $(NH_4)_2CO_3$ soln is added, and the precipitate filtered.</td></tr>
<tr><td>Group IV
White
($CaCO_3$, $SrCO_3$, $BaCO_3$)</td><td>Filtrate
Group V
1.Tested for Mg.
2.Evaporated to dryness with conc. HNO_3, flame tests for Na and K performed with residue.</td></tr>
</table>

****Removal of Interfering PO_4^{3-} radical using Zirconyl nitrate [$ZrO(NO_3)_2.2H_2O$]:**

H_2S is boiled off from the Group II filtrate. Solid NH_4Cl is added, dissolved and zirconyl nitrate solution added dropwise until precipitation is complete. The mixture is heated to boiling with stirring, and filtered. The residue is rejected and filtrate treated for Group IIIA.

B- Confirmatory Tests for Basic Radicals

Expeiment	Observation	Inference
Pb: White precipitate boiled with water to dissolve. To the solution acetic acid and K_2CrO_4 is added.	Crystalline white precipitate reappears on cooling. Yellow precipitate	Pb^{2+} confirmed
Bi: Black precipitate dissolved by boiling with dil. HNO_3 and excess conc. ammonia soln added, when white ppt forms.	White precipitate turns black with sodium stannite solution	Bi^{3+} confirmed

Cu: Black precipitate dissolved in dil. HNO_3, by boiling. Conc. NH_3 solution is added. Acetic acid and $K_4[Fe(CN)_6]$ soln added.	Deep blue solution Chocolate brown precipitate	Cu^{2+} confirmed
Cd: Yellow precipitate dissolved dissolved by boiling with dil. HNO_3 and excess conc. ammonia soln added. (i)Made strongly acidic with c. HCl, H_2S passed. (ii) Ammonia added, warmed, H_2S passed.	Blue solution Colourless solution (i)if black precipitate forms, it is filtered off. (ii)Yellow precipitate	Cu^{2+} present Cu^{2+} absent Cd^{2+} present
As: Yellow precipitate dissolved in $(NH_4)_2CO_3$ soln by boiling, then acidified with dil. HCl.	Yellow precipitate	As^{3+} confirmed
Sn: Yellow precipitate dissolved in conc. HCl by boiling, neutralised with Na_2CO_3, Fe-filings added and boiled, filtered into $HgCl_2$ soln.	White or grey precipitate	Sn^{2+} confirmed
Sb: Yellow precipitate dissolved by boiling with KOH solution, acidified with c. HCl and H_2S passed. Precipitate dissolved in c. HCl, made just alkaline with ammonia, solid oxalic acid added and H_2S passed.	Orange precipitate	Sb^{3+} confirmed
Fe: Reddish brown precipitate dissolved in dil. HCl, $K_4[Fe(CN)_6]$ soln added.	Prussian blue coloration /precipitate	Fe^{2+}/ Fe^{3+} confirmed
Mn: Brown precipitate dissolved in dil. HNO_3 by boiling, cooled, solid $NaBiO_3$ added, allowed to stand.	Violet/pink coloration	Mn^{2+} confirmed
Cr: Green precipitate boiled with NaOH and H_2O_2 soln. To the solution, acetic acid and lead acetate soln added.	Yellow solution Yellow precipitate	Cr^{3+} confirmed

Al: White precipitate dissolved in NaOH soln by boiling. Excess of solid NH_4Cl added and boiled.	White gelatinous precipitate	Al^{3+} confirmed
Ni: Black precipitate dissolved in aqua regia, evaporated till dry, residue dissolved in water. Excess NH4OH and dimethyl glyoxime is added.	Rose-red precipitate	Ni^{2+} confirmed
Co: Black precipitate dissolved in aqua regia, evaporated till dry, residue dissolved in water. Amyl alcohol and solid NH_4SCN added and shaken.	Blue coloration of organic layer.	Co^{2+} confirmed
Zn: White precipitate dissolved in dil. HCl, then excess NaOH soln added. Acidified with acetic acid and H_2S is passed.	White precipitate.	Zn^{2+} confirmed
Ba: White precipitate dissolved in hot dil. acetic acid. K_2CrO_4 soln added. Precipitate dissolved in conc. HCl, evaporated till dry, flame test performed with residue.	Yellow precipitate Apple green flame	Ba^{2+} confirmed
Sr: White precipitate dissolved in hot dil. acetic acid. Saturated $(NH_4)_2SO_4$ soln added, heated on water bath. Precipitate and filter paper charred in Bunsen flame, then flame test performed with residue.	White precipitate Crimson red flame	Sr^{2+} confirmed
Ca: White precipitate dissolved in hot dil. acetic acid and $(NH_4)_2C_2O_4$ soln added. Flame test performed with the precipitate.	White precipitate Brick-red flame	Ca^{2+} confirmed
Mg: To the solution (Group IV filtrate) NH_4Cl, NH_4OH and Na_2HPO_4 solution,	White crystalline precipitate	Mg^{2+} confirmed

shaken and inner walls scratched with a glass rod.		
Na: Aqueous extract of sample evaporated till dry and flame test performed with residue. To a neutral solution zinc uranyl acetate solution is added.	Golden yellow flame (colourless through cobalt double blue glass) Yellow precipitate or turbidity	Na^+ confirmed
K: Aqueous extract of sample evaporated till dry and flame test performed with residue. To a neutral solution sodium cobaltinitrite solution is added.	Lilac coloured flame (crimson through cobalt double blue glass) Yellow precipitate or turbidity	K^+ confirmed
NH_4^+: Sample boiled with NaOH and Nessler's Reagent added.	Orange precipitate	NH_4^+ confirmed

6. Analysis of Insoluble Compounds (insoluble in c. HCl and aqua regia)

Colour	Compound
Red	Fe_2O_3
Green	Cr_2O_3
White	Al_2O_3, CaF_2, $BaSO_4$, $SrSO_4$, SnO_2

<table>
<tr><td colspan="3">1.The solid is fused with a NaOH bead and KNO_3 on a mica foil, cooled, extracted with hot water and filtered.</td></tr>
<tr><td rowspan="2">Residue:
Dissolved in hot HCl, $K_4[Fe(CN)_6]$ solution added. Blue precipitate confirms Fe^{3+}.</td><td colspan="2">Filtrate: Divided into two parts. Yellow colour indicates chromium.</td></tr>
<tr><td>To 2 drops of the solution on a spot plate, 2 drops of Alizarin-S and acetic acid is added and rubbed with a glass rod. A violet colour, followed by a red precipitate confirms Al^{3+}.</td><td>To the yellow solution dil. acetic acid and $Pb(OAc)_2$ solution is added. Yellow precipitate confirms Cr^{3+}.</td></tr>
</table>

<table>
<tr><td colspan="4">2. The solid is fused with a NaOH bead and Na_2CO_3 on a mica foil, cooled, extracted with hot water and filtered.</td></tr>
<tr><td>Residue:
Washed with hot water and dissolved in acetic acid. Solution divided into three parts.</td><td colspan="3">Filtrate: acidified with c. HCl, boiled, divided into three parts.</td></tr>
<tr><td>1.To one part, K_2CrO_4 solution is added. Yellow precipitate, gives apple green flame on performing flame test with it. Ba^{2+} confirmed.
2. To another part, saturated $(NH_4)_2SO_4$ solution is added, boiled and filtered. White precipitate, gives crimson red flame Yellow precipitate, gives apple green flame on performing flame test with it. Sr^{2+} confirmed.
3. To the third part, NH_4OH and $(NH_4)_2C_2O_4$ solution is added and boiled. White precipitate, gives brick red flame on performing flame test with it. Ca^{2+} confirmed.</td><td>H_2S passed through the solution. Yellow precipitate confirms Sn^{4+}.</td><td>To the filtrate $BaCl_2$ solution is added. White precipitate confirms SO_4^{2-}.</td><td>To 2 drops of the solution on a spot plate, 2 drops of zirconyl nitrate-Alizarin-S reagent is added. Pink colour turning yellow confirms F^-.</td></tr>
</table>

7. Conclusion

The given sample contains:

Cation(s) --

Anion(s) —

1. Orientational Tests:

Sl. No.	Experiment	Observation	Inference
1	Sample heated in a dry test tube.	a. sample is yellow when hot, white when cold b. sample is yellow when hot and cold c. sample turns grey, brown, black or green d. white sublimate, remains white with H_2S, smell of NH_3 e. White sublimate, turns yellow with H_2S	a. Zn salt b. Pb salt c. Cu, Fe, Ni, Co, Cr salt d.NH_4^+ salts e. As salts
2	Flame test performed with sample, c. HCl and platinum wire.	a. golden yellow flame b. pinkish-violet (lilac) flame c. transient brick red flame d. apple green flame e. persistent crimson red flame f. bluish green flame	a. Na salt b. K salt c.Ca salt d. Ba salt e. Sr salt f. Cu salt
3	Borax bead test performed with a platinum wire loop, borax and the sample.	a. dark blue bead b. green bead, turns red on cooling c. violet (amethyst) bead d. yellow bead e. green bead	a. Co salt b. Cu salt c. Mn salt d. Fe salt e. Cr salt
4.	Sample fused on a mica foil with NaOH bead and KNO_3. The melt dissolved in distilled water, divided into 2 parts: To one part CH_3COOH, $Pb(CH_3COO)_2$ soln added. To other part CH_3COOH added.	a. yellow melt b. green melt a.yellow precipitate b. pink coloration	a. Cr salt b. Mn salt
5	Sample with Zn dust and d.HCl in a beaker stirred with a test tube half filled with water. The test tube is then held in the Bunsen flame.	Blue fluorescence in the outer wall of the test tube	Sn salt
6	Sample dissolved in d. HCl. Soln made ammoniacal with	Red precipitate	Ni salt

	NH_4OH, then DMG soln added.		
7	Sample dissolved in d. HCl. NaOH soln added till alkaline, boiled, H_2S passed.	White precipitate	Zn salt
8	Sample dissolved in d. NaOH. Solid NH_4Cl added, boiled, allowed to stand.	white gelatinous precipitate	Al salt
9	To a few drops of iodine-sodium azide soln in a watch glass solid sample is added.	Iodine soln decolorised	S^{2-}, SCN^- salts
10	Sample heated with d. H_2SO_4 [Test repeated with the addition of Zn dust]	a. colourless gas evolves with rotten egg smell, turns lead acetate paper shiny black. b. brown fumes evolve [colourless gas evolves with rotten egg smell, turns lead acetate paper shiny black.]	a.S^{2-} salts b. NO_2^- salts [covalent S^{2-} salts]
11	Sample heated with Na_2CO_3 and water.	pungent smell of ammonia	NH_4^+ salts
12	Sample heated with c. H_2SO_4.	a. brown fumes evolve and intensify on adding Cu turnings to test tube b. colourless gas forms dense white fumes with NH_3 c. reddish brown vapours d. violet vapours e. oily appearance of test tube, colourless gas which turns water drop(on glass rod) milky	a.NO_3^- salts b. Cl^- salts c. Br^-, BrO_3^- salts d. I^-, IO_3^- salts e. F^- salts
13	To the sample c. H_2SO_4 and methanol added, and evolved vapours ignited in Bunsen flame.	vapours burn with green flame at the mouth of the test tube.	BO_3^{3-} salts
14	Sampled boiled with c. HNO_3 and $(NH_4)_2MoO_4$.	canary yellow coloration	PO_4^{3-} , AsO_4^{3-} salts
15	(i)To aqueous soln or Na_2CO_3 extract of sample d. HCl and $BaCl_2$ soln added. (ii) To aqueous soln or Na_2CO_3 extract of	Heavy white precipitate insoluble in d. mineral acids. a. black precipitate b. yellow precipitate	SO_4^{2-} salts a.S^{2-} salts

	sample acetic acid and $Pb(CH_3COO)_2$ soln added.		b. CrO_4^{2-} /$Cr_2O_7^{2-}$ salts
16	To aqueous soln or Na_2CO_3 extract of sample d. HNO_3 and $AgNO_3$ soln added.	a.curdy white precipitate insoluble in d. HNO_3 but soluble in NH_4OH b. pale yellow precipitate partially soluble in NH_4OH c. yellow precipitate insoluble in NH_4OH	a.Cl^- salts b. Br^- salts c. I^- salts
17	To aqueous soln or Na_2CO_3 extract of sample d. HCl and $FeCl_3$ soln added.	blood red coloration	SCN^- salts
18	To sample soln in d. HCl, $K_4[Fe(CN)_6]$ soln added.	Prussian Blue coloration	Fe salts
19	To sample soln in d. HCl, excess NH_4OH added. To blue soln, CH_3COOH and $K_4[Fe(CN)_6]$ soln added.	Deep blue coloration Chocolate brown precipitate	Cu salts
20	To sample soln in d. HCl, amyl alcohol and solid $NH_4(SCN)$ added.	The organic layer turns blue	Co salts

Confirmatory Tests:

Sl. No.	Radical	Test	Observation
Cations:			
1	Na^+	Aqueous soln of sample evaporated to dryness, flame test preformed with the residue.	golden yellow flame, colourless through double cobalt blue glass.
2	K^+	Aqueous soln of sample evaporated to dryness, flame test preformed with the residue.	Violet (lilac) flame, crimson through double cobalt blue glass.
3	Ca^{2+}	To sample soln in d. HCl, acetic acid and $(NH_4)_2C_2O_4$ soln added. Flame test preformed with the precipitate.	White precipitate Brick red flame.
4	Sr^{2+}	To sample soln in d. HCl, acetic acid and saturated $(NH_4)_2SO_4$ soln added and heated. Precipitate and the filter paper charred in the flame, and Flame test preformed with the charred residue.	White precipitate Crimson flame.
5	Ba^{2+}	To sample soln in d. HCl, acetic acid and K_2CrO_4 soln added and boiled. Precipitate dissolved in c.HCl, evaporated to dryness, flame test preformed with the residue.	Yellow precipitate Apple green flame.
6	Al^{3+}	To sample soln in d. HCl, NH_4OH and excess NH_4Cl added. Precipitate dissolved in NaOH, boiled again with excess NH_4Cl.	Gelatinous white precipitate Precipitate reappears.
7	Cr^{3+}	To sample soln in d. HCl or water, NaOH and H_2O_2 added and boiled. To the soln CH_3COOH and $Pb(CH_3COO)_2$ soln added.	Yellow solution Yellow precipitate
8	Mn^{2+}	Sample soln in d. HNO_3 heated, cooled, and solid $NaBiO_3$ added.	Violet/pink coloration
9	Fe^{3+}	To sample soln in d. HCl or water, $(NH_4)SCN$ soln added.	Blood red colouration
10	Co^{2+}	To sample soln in d. HCl or water, amyl alcohol and solid $(NH_4)SCN$ added.	Blue coloration of organic layer.
11	Ni^{2+}	To sample soln in d. HCl or water, excess NH_3 and DMG soln added.	Red precipitate.
12	Cu^{2+}	To sample soln in d. HCl or water, excess NH_3 added. To the soln CH_3COOH and $K_4[Fe(CN)_6]$ soln added.	Deep blue coloration

			Chocolate brown precipitate.
13	Zn^{2+}	To sample soln in d. HCl or water, excess NaOH added and boiled. It is acidified with CH_3COOH and H_2S passed.	White precipitate.
14	Pb^{2+}	To sample soln in water d. HCl added. Precipitate dissolve in hot water and (i)cooled under the tap (ii)K_2CrO_4 soln added	White precipitate. (i)white crystalline precipitate (ii)yellow precipitate
15	Sn^{2+}	To sample soln in d. HCl or water, c.HCl and iron filings added and boiled. It is filtered into $HgCl_2$ soln.	White or grey precipitate.
16	NH_4^+	Aqueous soln of sample boiled with NaOH. Nessler's Reagent added.	Orange-brown precipitate.
Anions:			
17	F^-	Sample mixed with H_3BO_3 and c.H_2SO_4 to make a paste in a watch glass. A little of the paste is held close to the Bunsen flame with a glass rod.	Green flame coloration.
18	Cl^-	Solid sample, solid $K_2Cr_2O_7$ and c.H_2SO_4 heated in a test tube and the vapours passed into NaOH soln. CH_3COOH and $Pb(CH_3COO)_2$ soln added to the yellow soln.	Re vapours evolve which turn NaOH soln yellow. Yellow precipitate.
19	Br^-	To aqueous soln or Na_2CO_3 extract of sample, d.HCl, $CHCl_3$ and Cl_2-water is added.	Organic layer turns orange.
20	BrO_3^-	Solid sample, KBr and d.H_2SO_4 heated in a test tube.	Reddish brown vapours.
21	I^-	To aqueous soln or Na_2CO_3 extract of sample, d.HCl, $CHCl_3$ and Cl_2-water is added.	Organic layer turns violet.
22	IO_3^-	Solid sample, KI and d.H_2SO_4 heated in a test tube.	Violet vapours.
23	SCN^-	To aqueous soln or Na_2CO_3 extract of sample $FeCl_3$ soln added.	Blood red coloration.
24	S^{2-}	To aqueous soln or Na_2CO_3 extract of sample $Na_2[Fe(CN)_5NO]$ soln added.	Violet coloration.
25	SO_4^{2-}	To aqueous soln or Na_2CO_3 extract of sample d. HCl and $BaCl_2$ soln added.	Heavy white precipitate insoluble in mineral acids.
26	NO_3^-	To a pinch of the solid sample in a watch glass, one drop each of sulphanilic acid and α-naphthylamine, and Zn dust is added.	Red coloration

27	NO_2^-	To a pinch of the solid sample in a watch glass, one drop each of sulphanilic acid and α-naphthylamine, is added.	Red coloration
28	PO_4^{3-}	The sample is boiled with c. HNO_3 and $(NH_4)_2MoO_4$ soln. The test is repeated with the addition of tartaric acid.	Canary yellow precipitate obtained both times.
29	AsO_4^{3-}	The sample is boiled with c. HNO_3 and $(NH_4)_2MoO_4$ soln. The test is repeated with the addition of tartaric acid.	Canary yellow precipitate obtained only the first time.
30	BO_3^{3-}	Sample mixed with CaF_2 and c.H_2SO_4 to make a paste in a watch glass. A little of the paste is held close to the Bunsen flame with a glass rod.	Green flame coloration.
31	CrO_4^{2-}	To aqueous soln or Na_2CO_3 extract of sample CH_3COOH and $Pb(CH_3COO)_2$ soln added.	Yellow precipitate.