INDIAN SCHOOL MUSCAT
SENIOR SECTION
DEPARTMENT OF CHEMISTRY
CLASS XI-LAB SHEET

QUALITATIVE ANALYSIS OF INORGANIC SALT

Experiment Number. ---------- Date: ----------

Aim:

To identify the anion and cation present in the given salt systematically.

<table>
<tr><td colspan="5">PRELIMINARY EXAMINATION OF SAMPLE No: …….</td></tr>
<tr><td rowspan="6">1</td><td rowspan="6">Colour of the sample is noted</td><td colspan="2">The sample is:</td><td>Presence of:</td></tr>
<tr><td colspan="2">Blue</td><td>Cu^{2+} ions</td></tr>
<tr><td colspan="2">Green</td><td>Cu^{2+} or Ni^{2+} ions</td></tr>
<tr><td colspan="2">Dark yellow/Brown</td><td>Fe^{3+} or $Mn(NO_3)_2$</td></tr>
<tr><td colspan="2">Flesh or light pink</td><td>Mn^{2+} ions</td></tr>
<tr><td colspan="2">Yellowish brown</td><td>NH_4I salt</td></tr>
<tr><td rowspan="5">2</td><td rowspan="5">FLAME TEST: A <u>thick paste</u> of the sample is prepared in con. HCl and is shown to the non-luminous part of a Bunsen flame with the help of a glass rod.</td><td colspan="2">Colourless / White</td><td>Absence of transition metal cations.</td></tr>
<tr><td>Livid Blue flame</td><td>Presence of Pb^{2+} ion</td></tr>
<tr><td>Green-edged blue flame</td><td>Presence of Cu^{2+} ions.</td></tr>
<tr><td>Crimson flame</td><td>Presence of Sr^{2+} ions</td></tr>
<tr><td>Pale green flame</td><td>Presence of Ba^{2+} ions</td></tr>
<tr><td></td><td></td><td>Brick red flame</td><td>Presence of Ca^{2+} ion</td></tr>
</table>

ANALYSIS OF ANIONS

<table>
<tr><td>3</td><td>A little of the sample is treated with dil. HCl acid</td><td>Colourless, odourless gas (CO_2) is evolved with effervescence.</td><td>Presence of CO_3^{2-} ion
Note: Do the following test only if effervescence is seen in this test.</td></tr>
<tr><td>4</td><td>A little of the sample is treated with con. H_2SO_4.</td><td>White fuming, pungent smelling gas (HCl) produces dense white fumes when mouth of NH_4OH bottle is shown near the mouth of the test tube.
Note: Warm/Heat if necessary</td><td>Presence of Cl^- ion</td></tr>
<tr><td colspan="2"><u>Note: Difference in heating plays an important role in this test. Warming = Heat for 5 - 10 seconds</u></td><td>Reddish brown gas (NO_2) on <u>boiling</u> the mixture with copper turnings.</td><td>Presence of NO_3^- ion is confirmed.</td></tr>
<tr><td colspan="4">Confirmatory Tests for Carbonate(CO_3^{2-}) ion.</td></tr>
<tr><td></td><td>Pass the CO_2 gas through clear limewater.</td><td>Lime water turns milky</td><td>CO_3^{2-} ion is confirmed</td></tr>
<tr><td colspan="4"></td></tr>
<tr><td colspan="4">Confirmatory Tests for Chloride (Cl^-) ion.</td></tr>
</table>

	An aqueous solution of the sample is treated with dil. HNO_3 followed by $AgNO_3$ solution.	Thick curd like white (AgCl) precipitate dissolves in excess of NH_4OH solution.	Presence of Cl^- ion.

Confirmatory Test for Nitrate (NO_3^-) ion.			
	Treat an aqueous solution of the sample with dil. H_2SO_4, add freshly prepared $FeSO_4$ solution and agitate the mixture well. Now add con. H_2SO_4 to the mixture drop wise along the inside wall of the test tube.	A thin dark brown ring $\{[Fe(H_2O)_5NO]\}$forms at the junction between the reaction mixture and con.H_2SO_4.	Presence of NO_3^- ion is confirmed.
Note: ➢ ***Place the test tube containing the reaction mixture in beaker containing ~50 ml tap water and then only add con.H_2SO_4 to avoid temperature build up due to exothermic nature of the reaction.*** ➢ ***<u>Mixing ratio</u>: 1 ml of sample solution + 1ml dil.H_2SO_4 + 2 ml $FeSO_{4\,(aq)}$ and then add 1 ml of con. Sulphuric acid drop by drop.*** ➢ ***If precipitation takes during this test, add more dil.H_2SO_4 and then remove the precipitate by centrifugation.***			
Confirmatory Test for Sulphate (SO_4^{2-}) ion .			
An aqueous solution of the sample is acidified with **dil.HCl** followed by the addition of **$BaCl_2$** solution.		A white precipitate **($BaSO_4$)** formed, which is insoluble in **con.HCl**	Presence of SO_4^{2-} ion
An aqueous solution of the sample is acidified with dilute acetic acid followed by the addition of lead acetate [$(CH_3COO)_2$ Pb] solution.		A white precipitate ($PbSO_4$) formed, which is soluble in Ammonium acetate [CH_3 $COONH_4$] solution.	SO_4^{2-} ion is confirmed.

ANALYSIS OF CATIONS

Confirmatory Tests For NH_4^+ ion

Agitate a pinch of the solid salt sample with NaOH solution and note its smell. **(Note: Release the gas near your nose)**	Smell of ammonia (NH_3), which produces dense white fumes when Con. HCl is shown near the mouth of the test tube.	NH_4^+ ion confirmed.
The solid salt sample is treated with NaOH solution followed by Nessler's reagent. **Note: Do not shake the mixture.**	A brown precipitate is formed. (NH_2.HgO.HgI)	NH_4^+ ion confirmed.

INTER-GROUP SEPARATION OF CATIONS

A true solution of the salt is prepared in distilled water.
Note:
- **How to prepare? -** Start with a pinch of sample + 4ml of solvent. If it dissolves completely, then gradually increase the concentration by dissolving more salt.
- If the sample is insoluble in water; try dissolving it in dil.HCl or dil.HNO_3

	About 1 ml. of the sample solution is treated with dil. HCl	White precipitate of $PbCl_2$	Presence of group – I cations
	Through the above solution H_2S gas is passed.	Black precipitate of Copper sulphide (CuS)	Presence of group – II cations

Note:

- **H_2S WORK: Dirty H_2S gas delivery tube & its washing water can cause contamination of your work. Students should ensure that they are neat and clean before use.**
- **Reject any black precipitate obtained if the sample is not blue or green in colour**

	To about 2ml. (If the sample is insoluble in water; try dissolving it in dil.HCl or dil.HNO_3) of the salt solution **2 ml** of saturated NH_4Cl solution is added. Shake the mixture well and then add **7ml** of NH_4OH solution and agitate.	Gelatinous white precipitate of Aluminium hydroxide {$Al(OH)_3$}	Presence of group – III cations
		Brown precipitate Ferric hydroxide {$Fe(OH)_3$}	
Note: This solution may be called group-III solution			

	Through 2 ml of the of the above solution (group – III solution) H_2S gas is passed	Dirty white precipitate of Zinc sulphide (ZnS)	Presence of group – IV cations
		Buff precipitate of Manganese sulphide (MnS)	
		Black precipitate of Nickel sulphide (NiS)	
Note: Reject any black precipitate obtained if the sample is not green in colour			

	To 2ml of the group – III solution $(NH_4)_2CO_3$ (Little excess) solution is added	White precipitate of Barium carbonate ($BaCO_3$) Strontium carbonate (SrCO3) or Calcium carbonate ($CaCO_3$)	Presence of group – V cations

Confirmatory test for Magnesium (Mg^{2+}) ion

	To 2ml of the group –III solution Di-sodium hydrogen phosphate is added. (Note: Scratch the sides of the test tube with a glass rod if necessary)	White crystalline precipitate of Magnesium phosphate	Presence of Mg^{2+} ion confirmed
	2 Drops of salt solution are treated with 3 drops of Magneson reagent and 3 drops of NaOH solution	Blue precipitate / solution	Presence of Mg^{2+} ion confirmed

CONFIRMATORY TEST FOR CATIONS

ANALYSIS OF GROUP – I CATION			
Confirmatory Tests For Lead (Pb^{2+}) Ion			
	To one portion of the sample solution add few drops of Potassium chromate (K_2CrO_4) solution.	Yellow precipitate of Lead chromate	Presence of Pb^{2+} ion confirmed

		($PbCrO_4$)	
	To another portion of the sample solution add few drops of Potassium iodide (KI) solution.	Yellow precipitate of Lead iodide (PbI_2)	Presence of Pb^{2+} ion confirmed

ANALYSIS OF GROUP – II CATIONS			
Confirmatory Tests For Copper (Cu^{2+}) Ion			
	To one portion of the sample solution add excess of NH_4OH solution	Deep blue solution, $[Cu(NH_3)_4](OH)_2$	Presence of Cu^{2+} ion confirmed
	Treat one portion of the above deep blue solution with dil. Acetic acid followed by few drops of $K_4[Fe(CN)_6]$ (Potassium Ferro cyanide) solution	Chocolate coloured precipitate $Cu_2[Fe(CN)_6]$	Presence of Cu^{2+} ion confirmed

ANALYSIS OF GROUP – III CATIONS			
Confirmatory Tests For Aluminium (Al^{3+}) Ion			
	To one portion of the sample solution NaOH solution is added drop by drop.	White precipitate, $Al(OH)_3$, dissolves in excess of NaOH	Presence of Al^{3+} ion confirmed
	Another portion of the sample solution is acidified with dil.HCl followed by the addition of blue litmus solution. Shake the mixture well to get a pink coloured solution. To this solution NH_4OH solution is added drop wise without shaking.	A floating blue precipitate is formed (It is called the "Floating blue lake)	Presence of Al^{3+} ion confirmed

ANALYSIS OF GROUP – III CATIONS.			
Confirmatory Tests for Ferric (Fe^{3+}) Ion			
	To one part of the diluted salt solution $K_4[Fe(CN)_6]$ (Potassium ferr**o**cyanide) solution is added.	A Prussian blue colouration $Fe_4[Fe(CN)_6]_3$	Fe^{3+} ion confirmed.
	To 2nd part of the diluted salt solution Potassium thiocyanate (KCNS) solution is added.	A blood red colouration $Fe(CNS)_3$	Fe^{3+} ion confirmed.

ANALYSIS OF GROUP – IV CATIONS.			
Confirmatory Tests for Zinc (Zn^{2+}) Ion			
	To one portion of the sample solution NaOH solution is added drop wise without shaking.	White precipitate $\{Zn(OH)_2\}$ dissolves in excess of $NaOH_{(aq)}$	Zn^{2+} ion confirmed.
	To 2nd part of the sample solution $K_4[Fe(CN)_6]$ (Potassium Ferr**o** cyanide) solution is added.	Greenish/bluish white $[Zn_2[Fe(CN)_6]$ precipitate.	Zn^{2+} ion confirmed.

ANALYSIS OF GROUP – IV CATIONS.			
Confirmatory Tests for Manganese (Mn^{2+}) Ion.			
	To one portion of the sample solution add NaOH solution drop wise without shaking.	A white precipitate $\{Mn(OH)_2\}$ formed turns to brown (MnO_2)	Mn^{2+} ion confirmed.
	¼ spatula of solid sample is treated with ½ ml of con.HNO_3 followed by the addition of 1 ml of	A purple colour formed changes to brown on	Mn^{2+} ion confirmed.

	Sodium bismuthate solution[#.] **Note: Add water to get a colourless solution if needed**	standing.	
ANALYSIS OF GROUP – IV CATIONS.			
Confirmatory Tests for Nickel (Ni^{2+}) Ion. (Note: All salts containing Ni are Greenish in colour)			
	To one portion of the sample solution add 10 drops of Dimethyl glyoxime reagent followed by excess of NH_4OH solution.	A scarlet red precipitate.	Ni^{2+} ion confirmed
	To the 2nd portion of the sample solution add Br_2 water solution followed by NaOH solution, boil and then keep aside.	A black precipitate.	Ni^{2+} ion confirmed.

ANALYSIS OF GROUP V CATIONS			
Confirmatory Tests for Barium (Ba^{2+}) ion			
	To one portion of the sample solution add a few drops of Potassium chromate (K_2CrO_4) solution.	A yellow ($BaCrO_4$) "**precipitate".**	Ba^{2+} ion confirmed.
	To another portion of the sample solution add a few drops of Dil.H_2SO_4 acid. **Note:** ➢ **Do this test only if the K_2CrO_4 test is positive** ➢ **Do this test only as the 2nd confirmatory test for Ba^{2+}.**	A white ($BaSO_4$) precipitate.	Ba^{2+} ion confirmed.

ANALYSIS OF GROUP V CATIONS			
Confirmatory Tests for Strontium (Sr^{2+}) ion			
	A portion of the sample solution is treated with acetic acid followed by ammonium sulphate solution	Scanty white ($SrSO_4$) precipitate	Sr^{2+} ion confirmed.
	A ***thick paste*** of the sample is prepared in con. HCl and is shown to the non-luminous part of a Bunsen flame with the help of a glass rod.	Crimson coloured flame	Sr^{2+} ion confirmed.

ANALYSIS OF GROUP V CATIONS			
Confirmatory Tests Calcium (Ca^{2+}) ion.			
	To one portion of the sample solution add a few drops of Ammonium Oxalate {$(NH_4)_2C_2O_4$ } solution.	A white (CaC_2O_4) precipitate formed dissolves in Con. HCl.	Ca^{2+} ion confirmed.
	Another portion is made "***neutral"*** by adding NH_4OH followed by ***boiling*** till smell of NH_3 is gone. Cool this solution and then add 2 drops of NH_4Cl and few drops of $K_4[Fe(CN)_6]$ solution.	A white ($Ca(NH_4)K[Fe(CN)_6]$ precipitate. ***Note:*** ➢ ***Only a 'neutral' solution answers this test.*** ➢ ***If the medium is acidic or alkaline, the test fails.***	Ca^{2+} ion confirmed.

INDIAN SCHOOL MUSCAT
SENIOR SECTION
DEPARTMENT OF CHEMISTRY
CLASS XI-LAB SHEET

REPORT:

The given sample has: - Cation: ……..……., anion: ……..…….and the salt is … . . . ……..……

Note: Use only ionic and molecular formulae for reporting

NOTES & USEFUL INFORMATION:

1. Always record the analysis in a tabular form. (i. e. draw three columns for recording "Experiment', 'Observation' and 'Inference')
2. Give continuous serial numbers to tests while recording the analysis in your book.
3. **"*Notes*"** given in this 'analysis handout' are hints only, hence should not be written in the journal.
4. Some Manganese salts show sparking flame when flame test is conducted.
5. **"DANGER!"** Hot concentrated reagents should not be thrown into sink. {Cool the out side of the test tube to room temperature under running tap water and then carefully dilute the mixture by adding tap water little by little. Once the mixture is safely diluted, it may be washed normally.}
6. Whenever solubility of a precipitate formed is tested, a small amount (a **drop**) of the precipitate should be transferred to another test tube and dissolve in **excess** amount of solvent. (Always show two test tubes for attestation of the work)
7. ***When chloride ion is detected with Cu^{2+} or Mn^{2+} ions***, AgCl will not give a clear transparent solution with excess of NH_4OH (Solubility part of the 2nd confirmatory test). This may be avoided by washing the precipitate: [Centrifuge the mixture and discard the rest of the solution to separate AgCl. Add distilled water to the centrifuge tube, shake well to wash AgCl. Centrifuge again and discard water. Use the washed AgCl to do solubility test with NH_4OH.]
8. ***Nessler's reagent test*** should be conducted only after identifying the presence of NH_4^+ ion using NaOH test. (Zn^{2+} & Mn^{2+} ions can also give brown precipitate with Nessler's reagent)
9. Confirmatory tests for cations can also be done using true solutions made from the precipitate obtained during Inter-group separation of the cation in suitable solvents.
10. Only Green or Blue salts will give black precipitate with H_2S gas. If black precipitate is obtained with salts that are not blue or green it is due to contamination of the solution and should be rejected.
11. ***# → Sodium Bismuthate solution***: The reagent shows tendency to settle as it is sparingly soluble in distilled water. So whenever the reagent is used, do shake the reagent bottle well before transferring the reagent. Otherwise only supernatant water will get transferred to the test tube.
12. Second confirmatory test for Ni^{2+} ion may fail if Br_2 water used is weak.
13. ***If chloride ion is confirmed with Calcium ion*:** Neutralisation has to be compulsorily done as $CaCl_2$ shows acidic character.
14. ***2nd confirmatory test for Ca^{2+} ion*:** If the sample does not dissolve in distilled water, the true solution made should be properly neutralised as per the instruction given.
15. Write completed salt work in the Journal only after getting it corrected in the observation note book.