LAB MANUAL

B. Sc. I

AKHILESH SINGH

ASSISTANT PROFESSOR

DEPTT. OF CHEMISTRY

K. S. SAKET PG COLLEGE

AYODHYA

Laboratory Safety

1. A student should never stay in the laboratory alone or work without an instructor present.

2**.** Unanticipated results to an unauthorized or altered experiment can be quite hazardous. Materials should never be taken from the laboratory. Horseplay and pranks are never acceptable in the laboratory

3. Long hair and loose clothing that can get caught on glassware or otherwise be a hazard in lab should be pulled back.

4. Appropriate attire must be worn in lab**,** including shoes which cover the entire foot, and clothing which covers the legs and torso. Clothing items such as halter and midriff tops, as well as shorts, skirts and Capri pants, are inappropriate for laboratory work, as are sandals and flip-flops. Clothing worn should be capable of providing a barrier between your skin and the chemicals you are working with. Coverage of the lower legs and feet are necessary in case of dropped glassware.

5. Eating, drinking, smoking, chewing tobacco and applying cosmetics are forbidden in the laboratory**.**

6. All accidents and breakage must be reported to an instructor.

Any accident requires attention to clean up. Alert your neighbors immediately if there is a spill as to its nature and extent before you leave the area.

8. Pipetting by mouth suction is forbidden.

9. When needed, gloves must be worn. Gloves should be of a material and thickness appropriate for the reagents being used. However, gloves provide only a temporary layer of protection against chemicals on your skin and may be permeable to some chemical reagents, without visible deterioration. If your gloves come in contact with a chemical reagent, remove them, wash your hands, and get a new pair immediately. Hands must be washed just before leaving the laboratory.

10. All laboratory workers must know the location and proper use of all laboratory safety equipment**,** including eyewash, safety shower, fire extinguishers, and telephone.

Your instructor will show you the location of and proper use of laboratory safety equipment.

11. All laboratory workers must know how to safely evacuate the laboratory in the event of an emergency**.**

You should note all possible exits from your laboratory. Emergency classroom evacuation requires that the class reassemble away from the building to call roll and check for complete evacuation. Check with your instructor to confirm where the class should reassemble.

12. Items such as book bags, backpacks, purses and coats should be put out of the way off the

floor and lab bench.

13. Cell phones should be turned off before entering the laboratory.

14. Read labels and disposes of any waste in an appropriate waste container. Ask questions if you are not sure what to do.

15. Clean up in the laboratory is essential. Make sure you wash and put away all equipment before you leave the laboratory.

16. Inform your instructor at the beginning of the term if you have any special medical conditions that may need attention during the laboratory, such as known allergies or seizures.

Qualitative Inorganic Analysis

Systematic Analysis of Anion & Cations

S.No	Experiment	Observation	Inference
1	Preliminary Reactions	Colourless	Absence of Fe^{2+}, Fe^{3+}, Ni^{2+}, Co^{2+}.
	Appearance	Green Blue Brown Pink	May be Fe^{2+}, Ni^{2+}, Cu^{2+} Cu^{2+} May be Fe^{2+} May be Co^{2+}, Mn^{2+}
2	Action of heat Take a small amount of the given salt taken in a dry test tube, heat it gently; then strongly.	A colourless gas with a characteristic pungent odour turning moist red litmus paper blue.	May be NH_4^+ salt
		Reddish brown vapours turning acidified ferrous sulphate paper brown, are obtained.	May be
		Substance is white when cold and yellow when hot.	May be $(Zn)^{2+}$
3	Flame Test To a small amount of the given salt taken in a watch glass, add a drop of Con. HCl and make it into a paste. Introduce the paste with the help of a glass rod to the base of the non-luminous bunsen burner.	(i) Bluish green flame (ii) Apple green (iii) Brick red (iv) Crimson red	May be Cu^{2+} May be Ba^{2+} May be Ca^{2+} May be Sr^{2+}
Identification of Anions from Volatile Products:			
4	Action of dilute H_2SO_4: To a small portion of the given salt taken in a test tube add 1 or 2ccs of H_2SO_4 and gently warm it.	Brisk effervescence of colourless, odourless gas turning lime water, milky is obtained.	CO_3^{2-}
		Colourless gas with a smell of rotten eggs, turning lead acetate paper black is obtained.	sulphide.
		Colourless gas with smell of burning sulphur turning acidified dichromatic green is obtained.	SO_3^{2-}
		Reddish brown gas with fishy odour turning acidified ferrous sulphate brown is obtained.	
		Colourless gas with smell of vinegar is obtained.	CH_3COO^-

		No characteristic observation.	Absence of CO_3^{2-}, S^{2-}, SO_3^{2-} CH_3COO^-
5	Action of Conc. H_2SO_4 To a small amount of given salt taken in a test tube, add 2-3 ml of Conc. H_2SO_4 and gently heat it.	Reddish brown vapours turning moist fluorescent paper red.	Br^-
		Colourless gas with pungent smell giving dense white fumes with a glass rod dipped in NH_4OH solution.	Cl^-
		Violet coloured vapours turning starch paper blue or violet.	I^-
		Reddish brown vapours turning acidified ferrous sulphate paper brown.	NO_3^-
		No characteristic observation.	
6	Action of Conc. H_2SO_4 with Cu turnings: Mix a small amount of the given salt taken in a test tube with a few Cu bits, add 2 – 3 ccs of H_2SO_4 and heat it.	Copius evolution of reddish brown gas turning acidified ferrous sulphate paper brown is observed. No reddish brown vapours.	nitrate.
7	Action of Con.H_2SO_4 with MnO_2 To a small amount of the given salt taken in a test tube, add an equal amount of MnO_2 and add a few ccs of Con.H_2SO_4 and gently heat.	A greenish yellow gas turning starch iodide paper violet (or) blue is obtained.	chloride.
		Reddish brown vapours turning moist fluorescent paper red is obtained.	bromide.
		Violet vapours turning starch paper blue (or) violet is obtained.	iodide
		No characteristic coloured vapours are obtained.	
8	Action of NaOH To a pinch of the given salt taken in a test tube, add few ccs of 10% NaOH solution and gently warm it.	A colourless gas with a pungent smell giving dense white fumes with glass rod dipped in HCl is obtained. No characteristic gas is Ammonium liberated.	ammonium. Ammonium

Sodium Carbonate Extract

Identification of Anions in solution

Preparation of Extract:

Take about the given salt in a 100 ml beaker. Mix it well with about thrice its amount of solid sodium carbonate. Add about 15 – 20ml of distilled water. Mix well with neat glass rod. Boil contents of the beaker over Bunsen flame. Cool and filter through filter assembly. Collect the clean filtrate in another beaker. The Filtrate is called soda extract.

S.No	Experiment	Observation	Inference
9	Silver Nitrate Test: To a portion of extract add dilute HNO_3 until effervescence ceases. Add few drops in excess, 2 – 3nos of $AgNO_3$ solution.	Curdy white precipitate soluble in NH_4OH. Pale yellow precipitate sparingly soluble in NH_4OH. Yellow precipitate insoluble in NH_4OH. No precipitate is obtained.	Anion is Cl^- Anion is Br^- Anion is Absence of Br^-, Cl^-, I^-
10	Barium Chloride Test: To about one or two ccs of the extract, (after neutralizing with acetic acid and boiling of CO_2) add $BaCl_2$ solution. To a portion of the above ppt add dil. HCl.	A white precipitate insoluble in HCl. A white precipitate soluble in HCl. No precipitate is obtained.	
11	Lead Acetate Test: To about one or two ccs of the extract (after acidifying with acetic acid, boiling off CO_2 and cooling) add lead acetate solution.	White ppt, soluble in excess of ammonium acetate solution.	
12	Ferrous Sulphate Test: (Brown Ring Test) To about 1 or 2cc of extract add dilute H2SO4 in drops until the effervescence ceases. And few drops in excess add 2- 3 drops of freshly prepared $FeSO_4$ solution. Keeping the test tube in a slanting position, add Con. H_2SO_4 without disturbing the solution.	A brown ring is obtained at the junction of the liquid. No brown ring is observed.	Nitrate

13	Ferric Chloride Test: Take about 1 or 2ccs of the extract in a test tube and add neutral $FeCl_3$ solution. Filter, if required, and divide the solution or the filtrate in two parts: (i) To one part add dil.HCl (ii) To the second part add water and boil	Deep red colouration produced Red colouration disappears Reddish brown ppt.	
14	Calcium Chloride Test: To a portion of the sodium carbonate extract, taken in a test tube add dil. Acetic acid and boil off CO_2.Then add a few drops of calcium chloride solution. Add dil. HNO_3 to the white ppt and warm.	A white precipitate of calcium oxalate is obtained The precipitate dissolves.	Oxalate confirmed
15	Ethyl Acetate Test: To a pinch of given salt taken in a test tube, add a few drops of ethanol followed by 1 or 2ccs of H_2SO_4. Gently heat and cool it. Pour into Na_2CO_3.	A pleasant fruity odour is obtained.	acetate is confirmed.

Systematic Analysis of Cations

Preparation of Original solution:

Check solubility of the given salt in:

Cold water (or) hot water (or) Dil HCl (or) Conc. HCl. Label this solution as original solution.

Procedure for Separation of Basic Radicals into Groups

To the original solution, add Dil. HCl.

↓

<table>
<tr><td>White ppt. Group I present (Pb^{2+} and</td><td colspan="5"></td></tr>
<tr><td rowspan="6"></td><td colspan="5">If no ppt. pass H_2S through the given solution. If a coloured ppt. is formed, group 2 cations are present (Cu^{2+}), Pb^{2+}.</td></tr>
<tr><td rowspan="5"></td><td colspan="4">If no ppt is obtained from the above, boil off H_2s gas and add a few drops of conc. HNO_3 to the remaining solution. Cool, add 2-3g of solid NH_4Cl. Boil again and add NH_4OH solution till it becomes alkaline.</td></tr>
<tr><td colspan="4">If a ppt is formed, Group III cations are present. Reddish brown ppt. Fe^{3+} Gelatinous white ppt. Al^{3+}</td></tr>
<tr><td rowspan="4"></td><td colspan="3">If no ppt., pass H_2S to the given solution.</td></tr>
<tr><td colspan="3">If a ppt is formed Group IV cations are present. Black ppt. (Co^{2+}, Ni^{2+})
Flesh coloured ppt. Mn^{2+} white ppt. Zn^{2+}.</td></tr>
<tr><td rowspan="2"></td><td colspan="2">If no ppt is formed, boil off H_2S gas add $(NH_4)_2CO_3$ solution.</td></tr>
<tr><td colspan="2">If a white ppt is formed Group V cations are present (Ba^{2+}, Sr^{2+}, Ca^{2+})</td></tr>
<tr><td></td><td></td><td></td><td></td><td>If no ppt. Group VI cation is present (Mg^{2+}</td></tr>
</table>

If none of the cations are present, check for Group (0)

Cation Analysis

Group O (NH_4^+):

To a pinch of given salt add some water and warm. Then allow it to cool. Add Nessler's reagent and excess of NaOH solution.	Yellowish brown precipitate is obtained. No precipitate is obtained.	Ammonium. confirmed Ammonium. confirmed

Group I (lead):

Group I precipitate is dissolved by heating the precipitate with dil.HNO_3 or distilled water. Divide the solution into 3 portions and carry out the following reactions.

Experiment	Observation	Inference
To one portion of the above solution add dilute H_2SO_4.	A white precipitate of $PbSO_4$ is obtained.	Pb^{2+} is present.
To another portion, add potassium chromate solution.	A yellow precipitate of is obtained.	Pb^{2+}confirmed.
Golden Spangles Test: To the 3rd portion, add KI solution To above yellow precipitate, add some H_2O, boil and then cool.	A yellow precipitate is obtained. Precipitate dissolves and reappears in the form of golden spangles.	Pb^{2+} confirmed.

Group II

The group 2 precipitate is dissolved by heating with dilute HCl precipitate dissolves.

Experiment	Observation	Inference
Test for : To one portion of the above solution add drops of NH_4OH, until it is in excess.	A pale blue precipitate which dissolves in excess NH_4OH to give any inky blue solution is obtained.	Cu^{2+}confirmed.
To another portion, add dilute acetic acid and potassium ferrocyanide $[K_4(Fe(CN_6))]$	A chocolate brown ppt.	Copper confirmed

Group III:

Group III precipitate is heated with Conc. HCl and water, cooled and filtered.

Reddish brown precipitate indicates Fe^{2+} (or) Fe^{3+}

Gelatinous white indicates Al^{3+}

Experiment		Observation	Inference
1.	To 1 ml OS, add a few drops of dilute HCl and then add	Formation of a deep blue colour or ppt.	Fe^{2+} confirmed.
	0.5 cm^3 of potassium ferricyanide solution.		
2.	White gelatinous precipitate dissolves in minimum quantity of dil. HCl. To this, add a few drops of blue litmus solution. Add NH_4OH solution in excess.	Formation of a blue floating ppt. in colourless solution. (This is known as Lake test) A blue ppt., suspended in a colourless medium (called a lake)	Al^{3+} confirmed.
2B	To one part of the above solution add NaOH solution	White Precipitate soluble in excess of NaOH solution	Al^{3+} confirmed

3.	Reddish brown ppt. (Shows the presence of Fe^{3+}) Treat it with minimum quantity of dil. HCl solution to dissolve the ppt. and then heat. Divide the solution into two parts.	A yellow solution is produced.	Fe^{3+} may be
(i)	To one part, add few drops of potassium ferrocyanide solution.	A deep blue colour or ppt. is obtained.	
(ii)	To another portion add few drops of potassium sulphocyanide solution.	A deep red colour is obtained.	

Group IV :

Group IV precipitate is warmed with dilute HCl centrifugate

Black precipitate Ni^{2+}

Ni is not soluble in dilute HCl. To residue add Con.HCl, boil, cool. Divide the solution into two parts and boil of H_2S. To one part add dimethyl glyoxime reagent. Con.NH_4OH, till the solution becomes alikaline.	A Red rosy precipitate confirms the presence of Ni^{2+}
odium Hydroxide and Br_2 – water test. To the second part of the above solution, add NaOH and Br_2 water and then boil	A black precipitate confirms the presence of Ni^{2+}.
Confirmation of Zn^{2+}: Dissolve a part of white ppt in dil. HCl. Boil off H_2S and divide the solution in two parts. (i) To one part of solution add Pot. ferrocyanide solution (ii) To second part of solution add NaOH.	Bluish White ppt confirms Zinc. White ppt soluble in excess of NaOH is obtained.
Flesh (buff) colour precipitate indicates Mn^{2+}. Dissolve the precipitate in Dil. HCl and boil off H_2S then add NaOH solution.	A white precipitate is formed. Add Br water to the white ppt it turns black or brown. Mn^{2+} confirmed.

Lead peroxide test To the second part of the flesh coloured ppt, add a little of PbO_2 powder and ~~conc. HNO_3. Boil,~~ cool and allow to stand.	A pink colouration is produced. Mn^{2+} is confirmed.

Group V :

Take a small portion of Group V precipitate, carry out flame test. Take group V precipitate, add dilute CH_3COOH, warm. Boil of CO_2 and divide the solution into three parts.

Barium, Ba^{2+}	Stronium Sr^{2+}	Calcium Ca^{2+}
1. Potassium chromate test. To one part of the above solution, add a few drops of K_2CrO_4 solution – Yellow ppt. Ba^{2+} confirmed.	1. Ammonium sulphate test. To the second part of the above solution add a few drops of ammonium sulphate solution – white ppt. Sr^{2+} confirmed.	1. Ammonium oxalate test. To the third portion of the above solution, add ammonium oxalate solution and then NH_4OH solution to make it alkaline and scratch the sides of the test tube – white ppt. Ca^{2+} confirmed.
2. Dil. H_2SO_4 Test To another portion of the above solution, add a few drops of dil. H_2SO_4 – white ppt. insoluble in HCl. Ba^{2+} confirmed.	Flame test	Flame test

Test for Mg^{2+}:

1. Ammonium phosphate test. To the original salt solution, add solid NH_4Cl, warm to dissolve, cool and add NH_4OH solution in slight excess. Then add ammonium phosphate solution, shake well and allow to stand	White crystalline ppt.
2. To the original sat solution, add disodium hydrogen phosphate.	White crystalline ppt confirms Mg^{2+}

Additional Test:

Ash test for Al^{3+}, Zn^{2+}, Mg^{2+}

To a pinch of given salt taken in a test tube, add a few drops of Con.H_2SO_4, $Co(No_3)_2$. Mix it well. Dip one filter paper bit in a Bunsen flame. After it burns remove it from flame. After cooking observe the odour of the ash formed.	Blue tinted ash is obtained. Green tinted ash is obtained. Pink tinted ash is obtained.	Al^{3+}confirmed. Zn^{2+}confirmed. Mg^{2+}confirmed.

READY REFERENCE CHART FOR QUALITATIVE ANALYSIS OF BASIC RAIDCALS

<table>
<tr><td colspan="4">Make the solution of the given salt in distilled water, in dil. HCl or conc. HCl, cold or hot. Label it original solutions (O.S.). To the O.S., add dil. HCl</td></tr>
<tr><td rowspan="4">White ppt. Pb^{2+}
Confirmation of Pb^{2+}
Boil white ppt. with distilled water and divide the solution into two parts.</td><td colspan="3">If Group I is absent, pass H_2S gas through O.S. for sufficient time after acidification with dil. HCl filter.</td></tr>
<tr><td>PPt. - Group II
Black ppt. - (Pb^{2+} Cu^{2+})
Yellow ppt. - (As^{3+})
Dissolve the black ppt. in minimum quantity of 50% HNO_3. To one part</td><td colspan="2">If Group II is absent, add solid NH_4Cl, 0.5 ml conc. HNO_3 to the O.S. Boil and cool. Add NH_4OH till it smells of ammonia. If a ppt. is formed, group III is present.</td></tr>
<tr><td>Reddish brown ppt. - (Fe^{3+})
White ppt. - (Al^{3+})
Confirmation of (Fe^{3+})
Dissolve the brown ppt. in dil. HCl and divide</td><td colspan="2">If group III is absent, through a part of the solution obtained in group III pass H_2S gas. If a ppt. is obtained group IV is present.</td></tr>
<tr><td>White ppt. - (Zn^{2+})
Black ppt. - (Ni^{2+} and Co^{2+})
Flesh coloured ppt. - (Mn^{2+})</td><td>If Group IV is absent, to the O.S. add solid NH_4Cl, NH_4OH and $(NH_4)_2CO_3$ solution.</td></tr>
</table>

Add KI solution to one part of above solution. **Yellow ppt.** confirms Pb^{2+} .

Add K_2CrO_4 solution to the second part of the above solution. **Yellow ppt.** confirms Pb^{2+} .

of the above solution add. dil. H_2SO_4 and alcohol. If a white ppt. is formed Pb^{2+} is indicated. If no white ppt. is formed, add excess NH_4OH to the second part of the solution - Blue coloured solution indicates Cu^{2+} .

Confirmation of Cu^{2+}

Add excess of NH_4OH solution to original solution. **Deep blue solution.** Acidify the above deep blue solution with dil. acetic acid. Add **Pot. ferrocyanide solution. Chocolate brown ppt.** confirms Cu^{2+}.

the solution into two parts. To one part of solution add pot. ferrocyanide solution. A prussion blue colour or ppt. To second part of solution add. pot. sulphocyanide solution. A blood red colouration.

Confirmation of Al^{3+}

Perform charcoal cavity - cobalt nitrate test with white ppt. **Blue mass. Lake test.** White ppt. + dil. HCl + a few drops of blue litmus solution + NH_4OH solution in excess. **A blue ppt., suspended in a colourless medium** (called a lake)

Confirmation of Zn^{2+}

Dissolve a part of white ppt. in dil. HCl and divide the solution in two parts.

(i) To one part of solution add. pot. ferrocynaide solution - **Bluish white ppt.**

(ii) To second part of solution add NaOH solution. **White ppt. soluble in excess.**

Confirmation of Ni^{2+} and Co^{2+}

If the ppt. obtained is black Ni^{2+} or Co^{2+} is indicated. Note the colour of the salt. If the salt is greenish Ni^{2+} is indicated and if the salt is purple Co^{2+} is indicated.

Confirmation of Co^{2+}

(i) Take O.S. and neutralise the acid by adding NH_4OH. Add a pinch of **potassium nitrite** and acidify with dil. acetic acid - **Yellow ppt.**

(ii) Perform boraxbead test with the salt. - **Blue bead**

Confirmation of Ni^2

(i) Take O.S. and make alkaline by adding NH_4OH and then add a few drops of dimethyl glyoxime - **Bright red ppt.**

(ii) Perform borax bead test with the salt - **Brown bead** in oxidising flame and **grey bead** in reducing flame.

Confirmation of Mn^{2+}

(i) Dissolve flesh coloured ppt. in dil. HCl, boil off H_2S, add NaOH and Br_2 water. - **White ppt. turning grey.**

(ii) Perform borax bead test with group ppt. - **Pink bead** in oxidising flame and colourless in reducing flame.

White ppt. (Ba^{2+}, Sr^{2+}, Ca^{2+})

Dissolve a part of white ppt. in dil. acetic acid. Boil off CO_2.

To one part of the above solution, add **K_2CrO_4 solution. Yellow ppt.** Confirms Ba2+ .

If Ba^{2+} is absent, then to the second part of above solution add **amm. sulphate solution. A white ppt.** confirms Sr^{2+} .

If both Ba^{2+} and Sr^{2+} are absent, then to the third part of the above solution add **ammonium oxalate** solution. A **white ppt.** confirms Ca^{++} .

Perform the flame test with a part of the salt.

Light green flame-Ba^{2+} confirmed.

Crimson red flame-Sr^{2+} confirmed.

Brick red flame-Ca^{2+} confirmed.

If Group V is absent test for Mg^{2+} .

To the O.S. add a pinch of NH_4Cl, a few groups of NH_4OH and excess of **amm. phosphate** solution - **White ppt. – Mg^{2+} .**

Perform charcoal cavity cobalt nitrate test with white ppt. - **A pink mass.**

Test for NH_4^+

To the salt and sodium hydroxide solution and heat. If ammonia gas evolves NH_4^+ is present.

Pass the gas through **Nessler's reagent**. A **brownish ppt.** or colouration is obtained.

Note: NH_4^+ may be tested in the beginning of analysis of basic radicals as **zero group.**