EXPERIMENT-14

Qualitative analysis of a salt involving detection of one anion and one cation from the following (salts insoluble in hydrochloric acid excluded)

Anions :

(CO_3^{2-}, S^{2-}, NO_2^-, Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-}, PO_4^{3-})

Cations:

NH_4^+, Pb^{2+}, Cu^{2+}, Cd^{2+}, Fe^{3+}, Al^{3+}, Ni^{2+}, Zn^{2+}

Mn^{2+}, Ca^{2+},

14.1 OBJECTIVES

After performing this experiment, you should be able to:

- *explain the meaning of Qualitative Analysis;*
- *handle properly the chemicals and reagent bottles in the chemistry laboratory;*
- *perform simple experimental operations such as heating, testing of gas evolved, filtration and washing of residue etc.;*
- *observe the appearance / disappearance of colour, precipitate turbidity etc. in reaction mixture;*
- *acquire an elementary idea of the pressence of an anion or a cation present in the salt by carrying out dry test; and*
- *perform preliminary and confirmatory tests of anions and cations and analyse the given salt.*

14.2 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tubes	Hydrogen Sulphide gas
Test tube holder	
Funnel	**Solutions**
Glass rod	1) Lime water
Tripod stand	2) Silver nitrate Ag NO_3 (aq)
China dish	3) Lead acetate (aq)
Platinum wire	4) Sodium nitroprusside (aq)
Boiling tube	5) Potassium iodide (aq)
Brush	6) Starch solution
Beaker	7) Ammonium hydroxide
Wire gauze	8) Barium chloride (aq)
Spatula	9) Potassium chromate (aq)
Watch glass	10) Potassium ferrocyanide (aq)
	11) Potassium sulphocyanide
	12) Dimethyl glyoxime solution
	13) Ammonium oxalate (aq)
	14) Nesseler's reagent
	15) Blue litmus solution.
	16) Sodium hydroxide, (dilute and concentrated)
	17) Hydrochloric acid (dilute and concentrated)
	18) Sulphuric acid (dilute and concentrated)
	19) Nitric acid (dilute and concentrated)
	20) Acetic acid (dilute)
	Solids
	Ammonium chloride
	Ammonium carbonate
	Potassium dichromate
	Ferrous sulphate
	Ammonium Molybdate

14.3 WHAT YOU SHOULD KNOW

Qualitative analysis of an unknown salt consists of the detection and identification of the constituent ions. The inorganic salts on dissolving in water dissociate completely into positively and negetively changed ions. An positively charged ion is called *cation* or a basic radical and the negatively charged ion is called *an anion* or an acid radical. A number of tests are carried out to identify the ions.

Qualitative analysis is done by carrying out two kinds of tests (i) dry tests, and (ii) wet tests.

The dry tests are proformed on solid inorganic substances. These tests should be performed before proceeding with wet tests.

I. During a dry test, we note

(i) physical examination of salt such as colour, smell and density of the salt gives valuable clues regarding the nature of some of the basic and acid radicals.

(ii) action of heat on dry salt

II. The wet tests are carried out in solution. In a wet test, we note

(i) the colour and smell of the gas evolved.

(ii) formation or disappearance of a colour of solution.

(iii) formation or disappearance of a precipitate.

A test may be positive or negative. A positive test is the one which gives the result indicated in the theory and a negative test does not give the results as indicated in the theory.

For example, to test a carbonate ion, you add dilute sulphuric acid to the substance. The colourless gas evolved is passed through lime water which turns milky (a positive test). If the lime water does not turn milky, it is a negative test.

To detect the anion and cation in a salt, certain chemicals are used. These chemicals are called *Reagents*. When reagents react with salts, new compounds are formed with some observable properties such as colour, smell and characteristic appearance of precipitate.

Before carrying out systematic analysis, it is necessary to know few important laboratory techniques.

14.3.1 Laboratory Techniques

To identify an anion and a cation in a salt, some techniques have to be used. The techniques are as follows:

1. Heating of a salt or solution in a test tube

(i) While heating a salt or a solution in a test tube, hold the test tube in such a manner that the mouth of the test tube is away from yourself or any other person working in adjacent place.

(ii) Heat the test tube gently by placing its one side in the outermost zone of the flame. While heating, shake the test tube occassionally to avoid any spurtting.

(iii) Always heat the top layer of liquid in a test tube, so that it boils quickly. Never apply flame on the bottom of test tube, otherwise bumping will start. You may use a piece of procelein to avoid bumping.

(iv) While heating for a long time, use a test tube holder. Hold it between your thumb and the fingers if the volume in the test tube is less then half and you have to only warm the contents.

2. Use of a Reagent Bottle

Take out the desired reagent bottle from shelf. Remove the stopper and hold it in right hand. Hold the test tube between the thumb and first two fingers of the left hand. Now pour the required quantity of the reagent along the side of the test tube. Put the bottle back on the shelf and stopper it immediately. Never put the stopper on the table.

3. Testing of a gas evolved: On adding a reagent to a salt if effervescence are given out in cold or on heating it indicates the evolution of a gas.

The gas evolved can be tested in the following manner :

(i) Note the colour of gas evolved

(ii) Smell the evolved gas by puff of hand. Never inhale a gas by placing the nose directly into the vapour.

(iii) By bringing a filter paper strip or a glass rod with its end dipped into the testing reagent near the mouth of the test tube.

(iv) By passing the gas evolved for few seconds through a reagent taken in a test tube. shake the test tube well and observe the change.

4. Precipitation : Insoluble compound formed by addition of a reagent to a solution under the test or passing a gas through the solution under the test is called a precipitate and the process is called precipitation. As soon as a clear solution turns turbid, it is an indication that precipitate is formed.

In some cases the precipitate may dissolve in excess of precipitating reagent, in that case two observations should be taken, one on addition of precipitating reagent dropwise and second on adding excess of precipitating reagent.

5. Filtration : It is a process by which an insoluble compound is separated from a reaction mixture. To carry out filtration, first the filter paper cone is made. It is then opened in such a way that the three layers of paper are on one side, while the fourth layer is on the other side. By doing so a hollow cone of filter paper is obtained. The cone is then placed in funnel and by wetting with water, it is fixed properly but gently in funnel. The liquid mixture is then poured over the filter paper funnel along the side of a rod. Never fill the filter paper cone more than 2/3rd of its capacity. Note that there should be no space between filter paper cone and glass funnel. It reduces the rate of filtration. The liquid passes through the pores of filter paper and collects in the vessel kept below the stem of the funnel. The clear liquid so collected is called Filtrate. The insoluble compound or precipitate, which remains over the filter paper is called residue.

6. Washing and collection of precipitable : It is essential to wash the precepitate before carrying out any test on it. The washing is done by slowly pouring distilled water over the precepitate while it is still in the funnel. After washing. Carefully remove the filter paper from funnel open up and spread it on a dry filter paper and remove the precipitate carefully with the help of a spatula the precipitate carefully with the help of a spatula and collect it on a watch glass. Use a small portion of it eachtime for carrying out various tests.

7. Dry test: A number of tests can be carried out using the powdered salt. The information thus obtained often provides a clue to the presence or absence of certain radicals. With their knowledge, the course of wet analysis may be shortened or modified.

Some of the important dry tests are given below :

(i) Physical examination : Examine the colour, smell and the density of salt

S.No.	Observation	Inferences
1.	**The colour of solid**	
	(i) Blue	Copper salt may be present
	(ii) Green (light or dark)	Copper, nickel and ferrous salt may be present
	(iii) Yellow	Ferric salt may be present
	(iv) Pink	Manganese salt may be present
	(v) Buff	Manganese salt may be present
	(vi) White	Salts of remaining cations may be present (Ca^{2+}, Al^{3+}, NH_4^+, Pb^{2+}, Zn^{2+})
2.	**Smell :** Rub a pinch of salt with the help of a spatula on a watch glass smell of ammonia. smell of rotten eggs	 Ammonium salt may be present Sulphide salt may be present
3.	Density	
	Salt is light	Carbonates of aluminium, zinc and calcium may be present

(ii) Dry heating test : The salt is heated gently and then strongly in a clean and dry test tube.

S.No.	Observation	Inferences
(1)	Solid melts and resolidifies	Salts of calcium may be present
(2)	Solid swells up	Alum and phosphate may be present
(3)	Solid decripitates (Cracking sound)	Lead nitrate may be present
(4)	Solid sublies and vapour white in colour	Ammonium chloride may be present

14.3.2Precautions

(i) Use a perfectly dry test tube for performing this test.

(ii) Keep the mouth of the test tube away from yourself as well as from your neighbouring students.

(iii) During heating, do not heat the tube at one point but keep it rotating otherwise the tube may crack.

(iv) Do not smell the gases evolved by placing the nose directly over the mouth of the tube. Always smell the gas by puff of your hands.

14.4 DETECTION AND IDENTIFICATION OF ANIONS

List of anions : – CO_3^{2-}, S^{2-}, NO_2^- Cl^-, Br^-, I^-, NO_3^- SO_4^{2-} & PO_4^{3-}

14.4.1 Preliminary tests

The method of detection of anions is not as systematic as that of basic radicals. It has not been possible to include acidic radicals (anions) into distinct groups as in the case of cations (basic radicals).

The process employed to detect an anion may be divided into two classes.

(A) Those involving the identification of volatile products obtained on treatment with acids.

This test can be further divided into the following two groups,

(I) Action of dilute sulphuric acid

(II) Action of concentrated sulphuric acid.

(B) Those dependent on reaction in solution

Before carrying out a confirmatory test in solution it is necessary to prepare a water solution. Or sodium carbonate extract of the salt

(i) **Preparation of water solution for anion analysis :** Take a pinch of given salt in a test tube. Add 2=3 ml of water and shake well.

(ii) **Preparation of sodium carbonate of extract :** If the salt is insoluble in water, preparation of sodium carbonate extract is necessary.

This is prepared as follows :

Mix the salt with nearly twice its weight of sodium carbonate. Add sufficient distilled water in a dish, boil and filter (fig. 14.1(a) and (b). The filtrate is known as sodium extract.

How to use sodium carbonate extract : Sodium carbonate extract always contains an excess amount of unreacted sodium carbonate which may interfere in the usual tests of acid radicals. It is absolutely necessary to destroy the excess of sodium carbonate. This is done by acidifying sodium carbonate extract with a suitable acid (choice depends upon the nature of the acid radical to be tested).

(A) Action of Acids

(I) Test with dilute sulpuric acid : The anions which can be detected by treating a salt with dilute H_2SO_4, are as follows :

CO_3^{2-}, S^{2-} and NO_2^-

A gas is given out on adding dilute H_2SO_4 to salt. For observations and explanation follow the given table.

Anions	Observation	Explanation	Inferences
CO_3^{2-}	Brisk effervescence a colourless odourless gas is given out	$MCO_3 + H_2SO_4 \longrightarrow MSO_4 + H_2O + CO_2$ carbon dioxide (colourless)	CO_3^{2-} present
S^{2-}	Colourless gas with suffocating smell of rotten eggs is given out	$MS + H_2SO_4 \longrightarrow MSO_4 + H_2S$ Hydrogen sulphide (colourless)	S^{2-} may be present
NO_2^-	A brown coloured gas is evolved	$2MNO_2 + H_2SO_4 \longrightarrow M_2SO_4 + 2HNO_2$ nitrous acid (Colourless) $3\,MNO_2 \longrightarrow H_2O + HNO_3 + NO$ $NO + O_2 \longrightarrow 2NO_2$ Nitrogen dioxide (Reddish brown)	NO_2^- may be present

Do not boil the contents of the test tube. Over heating decomposes H_2SO_4 and gives sulphur dioxide.

(II) Confirmatory tests of CO_3^{2-}, S^{2-} and NO_2^- ions

(1) ***Carbonate ion*** CO_3^{2-}: On passing the gas through lime water, it turns milky due to the formation of calcium carbonate

$$Ca(OH)_2 + CO_2 \longrightarrow \underset{\text{Calcium carbonate}}{CaCO_3} + H_2O$$

On passing excess of CO_2, the milkiness disappears and a clear solution is obtained.

$$CaCO_3 + CO_2 + H_2O \longrightarrow Ca(HCO_3)_2$$

(2) ***Sulphide ion,*** S^{2-} :

(a) Filter paper moistened with lead acetate turns black on exposure to gas.

$$Pb(CH_3COO)_2 + H_2S \longrightarrow \underset{\text{Black lead sulphide}}{2CH_3COOH + PbS}$$

(b) Sodium Nitroprusside test : A violet colouration is obtained on adding freshly prepared solution of sodium nitroprusside to salt solution.

$$Na_2S + Na_2[Fe(NO)(CN)_5] \longrightarrow Na_4[Fe(CN)_5NOS]$$

Sodium Nitroprusside — Violet colouration

(3) Nitrite ion, NO_2 : When a filter paper soaked in a mixture of KI + starch solution is exposed to the gas, it turns blue or violet in colour.

$$2NO_2 + 2KI \longrightarrow 2KNO_2 + I_2$$

I_2 + Starch $\longrightarrow$ starch iodide complex, deep blue or violet.

(III) Test with concentrated sulphuric acid : This test is performed after performing the test with dilute H_2SO_4. Anions which give positive test with dilute H_2SO_4 will also react with concentrated H_2SO_4. On adding concentrated H_2SO_4 to a salt, if a gas is given out, follow the given table for drawing an inference.

I_{ON}	Observations	Explanation	Inferences
Cl^-	A colourless gas with pungent smell is evolved	$MCl + H_2SO_4 \rightarrow MHSO_4 + HCl$ Hydrogen Chloride (colourless gas)	Cl^- may be present

Br^-	A brown gas with pungent smell is evolved, the contents of the test solution turns reddish brown	$MBr + H_2SO_4 \rightarrow MHSO_4 + HBr$ $2HBr + H_2SO_4 \rightarrow 2H_2O + SO_2 + Br_2$ Bromine (reddish brown gas)	Br^- may be present
I^-	Violet fumes with pungent smell evolved Black specks appeared on the sides of the test tube.	$MI + H_2SO_4 \rightarrow MHSO_4 + HI$ $HI + 2H_2SO_4 \rightarrow 2H_2O + SO_2 + I_2$ [Iodine] purple (violet)	(I^-) Iodide, May be present
NO_3^-	brown coloured gas with pungent smell is evolved the gas intensified on adding copper turning	$2MNO_3 + H_2SO_4 \rightarrow M_2SO_4 + 2HNO_3$ $4HNO_3 \rightarrow 2H_2O + O_2 + 4NO_2$ Nitrogen dioxide (light brown gas) $3Cu + 8HNO_3 \rightarrow 2Cu(NO_3)_2 + 2NO + 4H_2O$ Nitrogen oxide (colourless) $2NO + O_2 \rightarrow 2NO_2$ Brown gas	NO_3^- may be present

Precautions

1. Do not boil the contents of test tube.
2. After the test is over, do not throw away the contents of test tube immediately into sink. The reaction of concentrated H_2SO_4 with water is highly exothermic and generate a lot of heat.
3. Do not inhale the gas evolved during the test because they are corrosive in nature.
4. Concentrated H_2SO_4 should be handled carefully.

(IV) Confirmatory tests of Cl^-, Br^- I^- and NO_2^-

(a) Chloride ion, (Cl^-)

(i) Silver Nitrate test : A curdy white precipitate insoluble in conc.HNO_3 is obtained on adding silver nitrate to water solution Acidify with dil HNO_3 before adding $AgNO_3$ in case sodium carbonate extract in used.

$$Ag^+ + Cl^- \rightarrow AgCl$$

Curdy white precipitate is soluble in ammonium hydroxide

$$AgCl + 2NH_4OH \longrightarrow [Ag(NH_3)_2]^+ Cl^- + 2H_2O$$

Diammine silver chloride (colourless)

(ii) Chromyl Chloride test–

Yellow fumes of Chromyl Chlorides are obtained on heating a mixture of salt and solid $K_2Cr_2O_7$ and concentrated H_2SO_4 in a dry test tube

$$2MCl_2 + K_2Cr_2O_7 + 3H_2SO_4 \longrightarrow 2MSO_4 + K_2SO_4 + 2CrO_2Cl_2 + 2H_2O$$

Chromyl chloride (orangish red)

Chromyl chloride fumes turns dilute NaOH yellow

$$CrO_2Cl_2 + 2\,NaOH \longrightarrow Na_2CrO_4 + 2HCl$$

Sodium chromate (yellow)

A yellow precipitate of lead chromate, $PbCrO_4$ is formed on adding lead acetate to the yellow solution acidified with CH_3COOH

$$Na_2CrO_4 + Pb(CH_3COO)_2 \longrightarrow PbCrO_4 + CH_3COONa$$

lead chromate (yellow precipitate)

(b) Bromide ion, Br^-

(i) Silver nitrate test : A light yellow precipitate is obtained on adding silver nitrate to water solution (after acidifying the sodium carbonate extract with dil HNO_3). The precipitate is partially soluble in ammonium hydroxide

$$Ag^+ + Br^- \longrightarrow AgBr$$

Silver bromide (light yellow)

$$AgBr + 2NH_4OH \longrightarrow [Ag(NH_3)_2]Br + 2H_2O$$

(ii) Organic layer test : The addition of chlorine water to water solution containing bromide liberates free bromine which imparts orange red colour to the test solution. On shaking with carbon tetrachloride, a reddish brown colour is imparted in CCl_4 layer. The organic layer of CCl_4 is obtained below the colourless aqueous layer.

$$2NaBr + Cl_2 \longrightarrow 2\,NaCl + Br_2$$

Bromine, reddish brown in CCl_4

(c) Iodide ion, I^-

(i) Silver nitrate test : Iodide ion, (I^-) A yellow precipitate is obtained on adding $AgNO_3$ solution to acidified (with dil HNO_3) water solution, which is insoluble in NH_4OH

$$Ag^+ + I^- \longrightarrow \underset{\text{Silver iodide, yellow precipitate}}{AgI}$$

(ii)Organic layer test : The addition of chlorine water to test solution liberates free iodine, which colours the solution, violet. On shaking with carbon tetra chloride, a violet colour is imparted to the organic layer.

$$2NaI + Cl_2 \longrightarrow 2NaCl + \underset{\text{Iodine (Violet Colour in } CCl_4)}{I_2}$$

(d) Nitrate ion, (NO_3^-) :

Ring test : On adding concentrated. H_2SO_4 gradually along the side of the test tube to a mixture of salt solution in water or its sodium carbonate extract, acidified with dil.H_2SO_4, and freshlyprepared ferrous sulphate solution, a brown ring is obtained at the junction of two layers.

$$M(NO_3)_2 + H_2SO_4 \longrightarrow MSO_4 + 2HNO_3$$

$$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + 4H_2O + 2NO$$

$$x\,FeSO_4 + y\,NO \longrightarrow \underset{\text{Nitrosoferrous sulphate, (brown ring)}}{(FeSO_4)_x(NO)_y}$$

(B) Individual tests

(a) Sulphate ion, SO_4^{2-}: On adding $BaCl_2$ Solution to acidified (dil HCl) salt solution or its sodium carbonate extract a white precipitate is obtained which is insoluble in concentrated HCl or concentrated HNO_3

$$Ba^{2+} + 2Cl^- \longrightarrow \underset{\text{Barium chloride white precipitate}}{BaCl_2}$$

(b) Phosphate ion, (PO_4^{3-}) : Salt or its water solution or its sodium carbonate extract is heated with concentrated. HNO_3 till the fumes no longer evolve. Contents of the test tube are diluted with water. A canary yellow precipitate of ammonium phosphomolybdate is formed on warming the above test solution with ammonium molybedate.

$$PO_4^{3-} + 12\,MoO_4^{2-} + 24\,H^+ + 3NH_4^+ \longrightarrow \underset{\text{Ammonium phosphomolybdate canary yellow precipitate}}{(NH_4)_3\,PO_4.12\,MoO_3.6H_2O}$$

Precautions

1. Some times on adding $BaCl_2$ Solution, a thin white precipitate is obtained, it may be due to formation of barium phosphate which is soluble in concentrated HCl.
2. During the test of phosphate ion, use a small porcelain piece or pumice stones while heating the salt or water solution with concentrated. HNO_3 to avoid bumping of contents of test tube.
3. Use conc HNO_3 carefully.

14.5 IDENTIFICATION OF CATIONS

For identification of cations (excluding NH_4^+ ion belonging to group zero) have been divided into six groups depending upon the differences in solubility of chlorides, sulphides, hydroxides and carbonates. Of these ions, the cations are precipitated by adding some reagents called, group reagents.

The following table gives brietly the classification of basic radicals (cations) into groups., Group reagents and form in which they precipitate.

Groups	Cations	Group reagent	Form in which cation precipitate
O	NH^+_4	Conc. NaOH	No precipitate obtanied. A Colourless gas ammonia with pungent smell is evolved on worning
I	Pb^{2+}	dil HCl	Chloride
II	Cu^{2+}, Cd^{2+}	H_2S(g) in the presence of dil HCl	Sulphide
III	Fe^{3+}, Al^{3+}	excess of NH_4OH in presence of excess of NH_4Cl (s)	Hydroxide
IV	Zn^{2+}, Mn^{2+}, Ni^{2+}	H_2S(g) in the presence af NH_4Cl(s) and, NH_4OH excess	Sulphide
V	Ca^{2+}	$(NH_4)_2CO_3$ soln. in the Presence of NH_4Cl and NH_4OH.	Carbonate

Before proceeding to detect the cations the solution of the salt should be prepared.

Preparation of original solution: Take a pinch of salt in a clean test tube. Add 2–3 mL of distilled water, shake well. If the solid is not soluble in cold water then heat the contents. If the salt is still not soluble in hot water add few drops of cone. HCl.

Precautions: Do not add excess of cone. HCl. This might create a problem during cation analysis.

(a) If on addition of conc. HCl a gas is evolved, wait till the effervescence ceases and then Add 2-3 drops more of conc. HCl.

(b) Use only distilled water to prepare original solution.

General rules for the procedure involving the analysis of cations (basic radicals)

(1) The group reagent should be added in the systematic order.

(2) Test for the higher group radicals are performed only when the radicals of a lower group are found absent.

(3) Slight excess of the group reagent should be added to ensure complete precipitation of group radicals.

(4) When a precipitate is obtained for a particular group, it is filtered and the residue is used for the analysis of the radicals of that group. Before carrying out the analysis, wash the residue two times with distilled water. Washings should be discarded.

(5) To find out the presence of a cation in a particular group, a small portion of the solution is used which is known as the test portion.

14.5.1 Cation Analysis

(a) Group zero, (Ammonium ion) NH_4^+

Ammonium ion, (NH_4^+) : On warming with NaOH solution, a colourless gas with pungent smell of ammonia is evolved.

$$NH_4^+ + OH^- \longrightarrow \underset{\text{Ammonia (colourless)}}{NH_3} + H_2O$$

Confirmatory test :

On passing the gas through Nessler reagent a reddish brown precipitate is obtained.

$$\underset{\text{Nessler's reagent}}{K_2HgI_4} \longrightarrow 2KI + HgI_2$$

$$HgI_2 + 2NH_3 \longrightarrow \underset{\text{(Amino-mercuric iodide)}}{Hg(NH_2)I} + NH_4I$$

$$2Hg(NH_2)I + 2H_2O \longrightarrow \underset{\text{Reddish brown, precipitate}}{\begin{matrix} Hg\langle_{O}^{NH_2} \\ Hg\langle_{I} \end{matrix}} + NH_4I$$

Iodide of millons base

(b) Group - I (Pb^{2+})

If a white precipitate is obtained on adding dilute HCl to salt solution in water or while preparing original solution in dilute HCl, it indicates the presence of Pb^{2+} ions. White precipitates dissolves again on warming and reprecipitates on cooling.

$$Pb^{2+} + 2Cl^- \longrightarrow PbCl_2$$

Lead chloride
(white precipitate)

Note : In case the original solution is prepared in dilute HCl, Group I is absent

(1) KI test : On adding KI to hot solution of lead chloride, a yellow precipitate is obtained.

$$Pb^{2+} + 2I^- \longrightarrow PbI_2$$

Lead iodide
(Yellow precipitate)

(2) Potassium chromate test : On adding K_2CrO_4 solution to hot– solution of lead chloride, a yellow precipitate is obtained.

$$Pb^{2+} + CrO_4^{2-} \longrightarrow PbCrO_4$$

Lead Chromate
(Yellow precipitate)

(C) Group II : (Cu^{2+} and Cd^{2+} ion)

On passing hydrogen sulphide gas through acidified original solution yellow or black precipitate is obtained.

Yellow precipitate indicates the presence of Cd^{2+} ions

Black precipitate indicates the presence of Cu^{2+} ions

$$M^{2+} + S^{2-} \longrightarrow MS$$

Metal sulphide

Note: Sometimes a precipitate is not obtained at group II level even if Cd^{2+} ions are present. Excess of H^+ ions prevents the precipitation of Cd^{2+} as CdS. To ensure the precipitation of Cd^{2+} ion, dilute a small portion of the solution and pass H_2S again.

(2) If no precipitate is obtained even on dilution of test portion, in that case gp II is absent.

(3) To obtain a granular precipitate pass H_2S for 2–3 minutes through hot solution.

Confirmatory test of Cu^{2+} ion : On heating black ppt of CuS with dilute HNO_3, a blue colour solution is obtained.

$$3CuS + 8HNO_3 \longrightarrow 3Cu(NO_3)_2 + 3S + 2NO + 4H_2O$$

Copper nitrate,
(blue colour)

(1) **On adding ammonium hydroxide, precipitate is obtained which is soluble in excess reagent to produce deep blue solution.**

$$Cu^{2+} + 2OH^- \longrightarrow \underset{\text{Copper hydroxide (Blue precipitate)}}{Cu(OH)_2}$$

$$Cu(OH)_2 + 4NH_3 \longrightarrow \underset{\text{Tetraammine cupric sulphate (deep blue colour)}}{[Cu(NH_3)_4]SO_4}$$

(2) **On adding acetic acid followed by potassium ferrocyanide to blue solution, a chocolate brown precipitate is obtained**

$$2Cu^{2+} + K_4[Fe(CN)_6] \longrightarrow \underset{\text{Copper ferrocyanide, (chocolate brown precipitate)}}{Cu_2[Fe(CN)_6] + 4K^+}$$

Confirmatory test of Cd^{2+} ion : On heating yellow precipitate (CdS) with dilute nitric acid a colourless solution is obtained.

$$3CdS + 8HNO_3 \longrightarrow \underset{\text{Cadmium nitrate (colourless)}}{3Cd(NO_3)_2} + 4H_2O + 2NO + 3S$$

On adding NH_4OH to the above obtained solution, a white precipitate is obtained, which dissolves in excess of reagent

$$Cd^{2+} + 2OH^- \longrightarrow \underset{\text{Cadmium hydroxide, (white precipitate)}}{Cd(OH)_2}$$

$$Cd(OH)_2 + 4NH_3 \longrightarrow \underset{\text{Tetra-amminecadmium hydroxide (colourless)}}{[Cd(NH_3)_4](OH)_2}$$

On passing hydrogen sulphide gas through above solution a yellow precipitate of CdS reappears.

$$[Cd(NH_3)_4](OH)_2 + H_2S \longrightarrow \underset{\text{Cadmium sulphide (yellow precipitate)}}{CdS} + 4NH_3 + 2H_2O$$

(d) Group III

(Fe^{3+} and Al^{3+} ions)

Preliminary test: To the origional solution add little concentrated HNO_3 and boil to oxidise Fe^{2+} to Fe^{3+} and then add excess of solid NH_4Cl followed by excess of ammonium hydroxide. White or reddish brown precipitate is obtained.

Reddish brown precipitate indicates the presence of Fe^{3+} ions.

White gelatinous precipitate indicates the presence of Al^{3+} ion

$$M^{3+} + 3OH^- \longrightarrow M(OH)_3$$

Metal hydroxide

Note : Addition of large excess of NH_4OH may start dissolving group III precipitates. Hence it should be avoided.

Confirmatory test of Fe^{3+} ion : Reddish brown precipitate of $Fe(OH)_3$ on dissolving in dilute HCl, produces a yellow solution

$$Fe(OH)_3 + 3HCl \longrightarrow \underset{\text{Yellow solution}}{FeCl_3} + 3H_2O$$

(1) On adding $K_4[Fe(CN)_6]$ to yellow solution, a prussian blue precipitate is obtained.

$$4Fe^{3+} + 3K_4[Fe(CN)_6] \longrightarrow \underset{\text{Prussian blue}}{Fe_4[Fe(CN)_6]_3} + 12K^+$$

(2) On adding KCNS to yellow solution a red colouration is obtained

$$Fe^{3+} + 3SCN^- \longrightarrow \underset{\text{Ferric thiocyanate (red colour)}}{[Fe(SCN)_3]}$$

Confirmatory test of Al^{3+} ion white gelatinous precipitate of $Al(OH)_3$ soluble in dilute HCl.

$$Al(OH)_3 + 3HCl \longrightarrow \underset{\text{Aluminium chloride colourless}}{AlCl_3} + 3H_2O$$

Blue Lake test : On adding blue litmus solution to above solution a red colouration is obtained. On adding NH_4OH drop by drop till the solution is alkaline a blue floating precipitate is obtained.

$$Al^{3+} + 3NH_4OH \longrightarrow Al(OH)_3 + 3NH_4^+$$

Aluminium hydroxide $Al(OH)_3$ precipitate adsorbs blue litmus.

(e) Group IV analysis (Zn^{2+}, Mn^{2+} & Ni^{2+} ions)

Preliminary test : Take the original solution and add NH_4OH to make it alkaline pass hydrogen sulphide, gas (H_2S) through the solution, a precipitate is obtained

Formation of white precipitate indicates the presence of Zn^{2+}

Formation of bulf (flesh coloured) precipitate indicates the presence of Mn^{2+}

Formation of black precipitate indicates the presence of Ni^{2+}

$$M^{2+} + S^{2-} \longrightarrow \underset{\text{Metal sulphide}}{MS}$$

Note : Hydrogen sulphide gas should always be passed slowly, Otherwise black residue of FeS from kipps appratus enters the test solution and creates confusion.

(2) Solution should smell of ammonia before passing H_2S.

Confirmatory test: Zn^{2+} ion

On dissolving white precipitate of ZnS. in dilute HCl, a colourless solution is obtained Boil off H_2S.

$$ZnS + 2HCl \longrightarrow ZnCl_2 + H_2S$$

(1) On adding small amount of NaOH a white precipitate is obtained which is soluble in excess of NaOH

$$Zn^{2+} + 2OH \longrightarrow Zn(OH)_2$$

Zinc hydroxide,
(white precipitate)

$$Zn(OH)_2 + 2OH^- \longrightarrow [Zn(OH)_4]^{2-}$$

Tetrahydroxozincate (II) ion

(2) Gray white precipitate is obtained on adding potassium ferrocyanide to solution of ZnS in dilute HCl.

$$2Zn^{2+} + K_4[Fe(CN)_6] \longrightarrow Zn_2[Fe(CN)_6] + 4K^+$$

grey white precipitate

Confirmatory test of Mn^{2+} Bulf coloured precipitate of MnS dissolves in dilute HCl.

$$MnS + 2HCl \longrightarrow MnCl_2 + H_2S$$

Boil off hydrogen sulphide gas. To the solution obtained above, on adding NaOH, a white precipitate of $Mn(OH)_2$ is obtained. The precipitate rapidly oxidises on exposure to air turning brown.

$$Mn^{2+} + 2OH^- \longrightarrow Mn(OH)_2$$

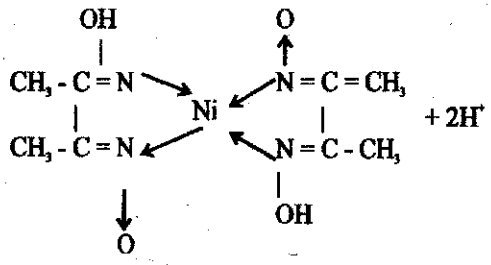
Manganese hydroxide
(white precipitate)

$$Mn(OH)_2 + O_2 \longrightarrow MnO_2 . H_2O$$

Hydrated Manganese dioxide,
(brown precipitate)

Confirmatory test : *Ni^{2+} ion* – Black precipitate of NiS is soluble in hot conc. HNO_3. giving light green solution. On adding NH_4OH and dimethylglyoxime reagent to above solution, a cherry red precipitate of nickel dimethyl glyoxime is obtained.

$$3\,NiS + 2HNO_3 + 6H^+ \rightarrow 3Ni^{2+} + 2NO + 4H_2O + 3\,S$$



Nickel dimethylgly oxime
Cherry red precipitate

(b) Group V (Ca^{2+} ion)

Preliminary test : A pinch of NH_4Cl is added to original solution. On adding excess of NH_4OH to make the solution alkaline then add freshly prepared $(NH_4)_2CO_3$ solution. a white precipitate is obtained

$$M^{2+} + CO_3^{2-} \longrightarrow MCO_3$$

Metal carbonate
(white precipitate)

White precipitate is soluble in acetic acid

$$MCO_3 + 2CH_3COOH \longrightarrow M(CH_3COO)_2 + H_2O$$

Metal acetate
(Colourless)

A white precipitate of calcium oxalate is formed on adding ammonium oxalate to above solution.

$$Ca(CH_3COO)_2 + (NH_4)_2C_2O_4 \longrightarrow CaC_2O_4 + 2CH_3COONH_4$$

Calcium oxalate
(white precipitate)

14.6 HOW TO PERFORM THE EXPERIMENT

Tabular presentation of qualitative test

14.6.1 Acid Radicals

(i) Work sheet No.-1 Carbonate ion (CO_3^{2-}), Sulphide ion (S^{2-}) and Nitrite ion (NO_2)

Experiment	Observation	Inference
(1) **Carbonate ion, CO_3^{2-}** Take a small alt in a clean test tube. Add 1 ml of dilute H_2SO_4.	**Brisk effervescence** Colourless, odourless,	**Carbonate, (CO_3^{2-}) may be present**
Lime water test Pass the gas evolved through 2–3 ml of lime water and shake well	Lime water turns milky	CO_3^{2-} confirmed
(2) **Sulphide ion, (S^{2-})** Take a small amount of given salt in a clean test tube. Add 1 ml of dil H_2SO_4.	Colourless gas with suffocating smell of rotten egg is given out	Sulphide, S^{2-} may be present
(i) Confirmative test : Bring a piece of filter paper moistened with lead acetate solution near the mouth of test tube	**Paper turns black**	**Sulphide, S^{2-} confirmed**
(ii) Sodium nitroprusside test: To 2 ml of Water solution of salt add 1 ml of freshly prepared sodium nitroprusside solution Shake well	A red colouration is obtained	Sulphide (S^{2-}) ion, confirmed
(3) **Nitrite ion, NO_2^-** Take a small amount of give salt in a clean test tube. Add 1 ml of dilute H_2SO_4.	A brown coloured gas with pungent smell is given out	Nitrite, (NO_2^-) may be present
Test the gas evolved in Preliminary test with a filter paper soaked in mixture of KI and starch solution	Blue black or violet colour is obtained	Nitrite, (NO_2^-) ion, confirmed

(ii) Work sheet No. 2 : Chloride ion (Cl^-), Bromide ion (Br^-), Iodide ion (I^-) and Nitrate ion NO_3^-

Experiment	Observations	Inference
(A) Chloride ion, (Cl^-) Take a small amount of given salt in a clean and dry test tube. Add 2=3 drops of conc. H_2SO_4 carefully. Heat the test tube gently.	A Colourless gas with pungent smell is evolved is evolved	Chloride ion, (Cl^-) may be present
(I) **Chromyl Chloride test :** Take a pinch of salt and solid potassium dichromate in a ratio of 1 : 3 in a dry test tube. Add 3–4 drops of concentrated H_2SO_4 and heat strongly.	orange – yellow fumes are given are out	
(ii) Pass the gas evolved through NaOH solution. Shake well.	A yellow solution is obtained	
(iii) Acidify the soln. obtained above with acetic and neutralize with excess of NaOH and then add lead acetate solution to it. Shake well.	A yellow precipitate is obtained	Cl^- Confirmed
(2) **Silver nitrate test :** (i) To 5 mL of original solution dil HNO_3 to acidify it and then add 1 mL of $AgNO_3$ solution. Shake well.	Curdy white precipitate is obtained	Cl^- may be present
(ii) Filter the precipitate. After washing the precipitate add 2=3 ml of ammonium hydroxide solution to it. Shake well.	A colourless solution is obtained	Cl^- Confirmed
(B) Bromide ion, (Br^-) Take a small amount of given salt in a clean and dry test tube Add 2–3 drops of concentrated H_2SO_4 . Heat the test tube carefully.	A brown gas with pungent smell is evolved. The contents of the test tube turns orange red	Bromide ion (Br^-) may be present
(i) **$AgNO_3$ test:** Acidify 2ml of original soln with dilute HNO_3 Add silver nitrate solution.	A light yellow precipitate is obtained which is partially soluble in excess of NH_4OH	Bromide ion, (Br^-) confirmed

(ii) **Organic layer test:** To 2 mL of water solution add 1/2 mL of CCl_4 and add 3 mL of Chlorine water. Shake well.	Reddish brown organic layer is obtained	Bromide ion, (Br^-) confirmed
(C) **Iodide ion, I^-** Take a small amount of given salt in a clean and dry test tube Add 2=3 drops of concentrated H_2SO_4 carefully. Heat the test tube gently.	Violet fumes with pungent smell evolved Black specks appeared on the side of the test tube.	Iodide ion, (I^-) may be present
(i) **$AgNO_3$ test:** To 2 mL of water solution add dilute HNO_3 to acidify it add 1 mL at $AgNO_3$ solution. shake well.	A yellow precipitate is obtained	
(ii) Add NH_4OH to the above solution ppt add 2–3 mL of ammonium hydroxide solution to it. Shake well	precipitate is insoluble	Iodide ion (I^-) confirmed
Take 2 mL of water solution. add 1/2 mL of CCl_4. Then add 3=4 mL of Chlorine water. Shake well.	A violet organic layer is obtained	I^- confirmed
(D) **Nitrate ion, NO_3^-** Take a small amount of given salt in a clean and dry test tube Add 2–3 drops of concentrated H_2SO_4 carefully. Heat the test tube gently.	Brown coloured gas with pungent smell is evolved. The gas intensified on adding copper turnings, solution turns blue.	nitrate ion, (NO_3^-) may be present
Take 2–3 ml of water solution in a test tube and add 2 mL of freshly prepared solution of ferrous sulphate, shake well. To this solution add few drops of concentrated H_2SO_4 gradually along the sides of test tube without disturbing the test tube.	A brown ring is obtained at the junction where the two liquid surface meet each other.	Nitrate ion, (NO_3^-) confirmed

(iii) Work Sheet No.3 : Qualitative analysis of Sulphate ion (SO_4^{2-}) and Phosphate ion (PO_4^{3-}) ion

Experiment	Observation	Inference
(A) Sulphate ion, (SO_4^{2-})		
(i) Acidify the Na_2CO_3 extract or the salt solution in water with dil.HCL and then add $BaCL_2$ solution.	A whilte ppt insoluble in Conc.HCl is obtained.	SO_4^{2-} is confirmed
(B) Phosphate ion, (PO_4^{3-}) Acidify the Na_2CO_3 extract in the salt solution in water with dil. HNO_3 and then add solid ammounium emolybdete. Warm the test tube gently.	A canary yellow precipitate or colour is obtained	Phosphate ion (PO_4^{3-}) ion confirmed.

14.6.2 Basic Radicals

(i) Work sheet No. 1: Ammonium ion (NH_4^+)

Experiment	Observation	Inference
Take a pinch of salt in a test tube. Add 2–3 ml. of NaOH solution. Heat the test tube gently and then strongly (if necessary) smell the gas evolved.	A colourless gas with pungent smell is given out.	Ammonium ion (NH_4^+) may be present.
Pass the gas evolved in the above test through Nessler's reagent for alteast 1 minute. Shake well.	A reddish brown precipitate is obtained.	Ammonium ion, (NH_4) confirmed.

(ii) Work Sheet No. 2 : Group- I : Lead (II) ion (Pb^{2+})

Experiment	Observation	Inference
Solubility in water Take a pinch of salt in clean test tube. Add 2–3 ml of water and then few drops of dilute HCl. Shake well.	A white precipitate is obtained.	Lead (II) ion (Pb^{2+}) may be present.
Carry out following tests with white precipitate obtained above.		

Wash the precipitate and dissolve a small amount of precipitate in 5–6 ml of hot water. Divide the soln. into two parts.	A clear solution is obtained.	
(a) **Lead iodide test :** To one part of hot soln. add 1 ml lead iodide solution. shake well.	A yellow precipitate is obtained.	Lead (II) ion (Pb^{2+}) confirmed.
(b) **Potassium Chromate test** To the second part of hot solution add 1 ml of K_2CrO_4 solution and shake well.	A yellow precipitate is obtained.	Lead (II) ion (Pb^{2+}) confirmed.

(iii) Work Sheet No. 3 : Group II Copper (II) ion (Cu^{2+}) and cadmium (II) ion (Cd^{2+}).

Experiment	Observation	Inference
Take 2 ml of original solution in a test tube, add a few drops of dilute HCl. Pass H_2S gas through this solution.	(i) black precipitate (ii) yellow precipitate	(i) Copper (II) ion (Cu^{2+}) may be present. (ii)Cadmium ion (Cd^{2+}) may be present.
(a) In case a black precipitate is obtained carry out following tests:	A black precipitate is obtained.	Copper (II) ion, Cu^{2+} may be present.
Heat a small amount of precipitate in 1–2 ml of dilute HNO_3 and divide it into two parts.	A blue solution is obtained	Copper (II) ion, (Cu^{2+}) may be present.
(i) To one part add NH_4OH solution drop by drop till it is in excess.	A blue precipitate is obtained which dissolves to produce deep blue solution.	Copper (II) ion, (Cu^{2+}) confirmed.
(ii) Acidify another portion with 2 ml acetic acid and then add 1 ml of potassium ferrocyanide.	A chocolate brown precipitate is obtained.	Copper (II) ion Cu^{2+} ion confirmed.
Confirmatory test of Cadmium (II) ion Cd^{2+} : In case yellow precipitate is obtained group II carry out following tests.	A yellow precipitate is obtained.	Cadmium (II) ion Cd^{2+} may be present.
Test: (i) Take a small amount of the precipitate in a clean test tube add 1 ml of distilled water and 1 ml of dilute HNO_3. Heat strongly.	A colourless solution is obtained.	

(ii) To the above solution add NH_4OH solution drop by drop till it is in excess.	A white precipitate is obtained which dissolve in excess to produce colourless solution.	
(ii) Pass H_2S gas through above obtained solution.	A yellow precipitate is obtained	Cadmium (II) ion Cd^{2+} Confirmed.

(iv) Work Sheet No. 4 : Group III Aluminium (III)ion, (Al^{3+}) and Ferric iron or Iron (III) ion, (Fe^{3+})

Experiment	Observation	Inference
Take 2 ml of original solution in a clean test tube. Add excess of NH_4Cl solid, shake well to dissolve it. Heat if necessary. To cold solution add NH_4OH drop by drop till it is in excess.		
(A) **Aluminium ion, (Al^{3+})** : If a white gelatinous precipitate is obtained, it indicates the presence of Al^{3+} ion.	A white gelatinous precipitate obtained.	Aluminium (Al^{3+}) may be present.
Blue lake test: Dissolve a small amount of precipitate in 1 ml of dilute HCl. Add 2–3 drops of blue litmus solution. Shake well. Then add NH_4OH solution drop by drop until the solution is just alkaline.	A clear soln. is obtained A red colour solution is obtained A blue floating precipitate is obtained.	Aluminium (III) ion, (Al^{3+}) ion confirmed.
(B) **Iron (III) ion, (Fe^{3+})** If a reddish brown precipitate is obtained, it indicates the presence of Fe^{3+} ion.	A reddish brown precipitate obtained.	Fe^{3+} ion may be present.
Dissolve the precipitate in 2–3 ml of dilute HCl. and divide it into two parts.	A yellow solution is obtained.	
(i) To one part of solution add 1 ml of potassium ferrocyanide. Shake well.	A prussian blue colour is obtained.	Iron (iii) ion Fe^{3+} confirmed.
(ii) To the second part of solution add 1 ml of potassium sulphocyanide.	A red colouration is obtained.	Iron (III) (Fe^{3+}) ion confirmed.

(v) Work Sheet No. 5 : Group IV-Zinc (II) ion (Zn^{2+}), Manganese (II) ion (Mn^{2+}), and Nickel (II) ion (Ni^{2+})

Experiment	Observation	Inference
Take a 2–3 ml of original solution in a test tube, add excess of NH_4OH to make the solution alkalline. Warm the contents of the test tube. Pass H_2S gas through it for 1/2 minute		
(A) **Zinc (II) ion, Zn^{2+}**: Formation of white or dirty white ppt indicates the presence of Zn^{2+} ions.	White precipitate is obtained.	Zinc (II) ion (Zn^{2+}) may be present.
Dissolve the washed white precipitate in 1mL of dilute HCl. Dilute it with 2–3 ml of water. Divide the solution into two parts.	A clear soln. is obtained.	
To one part of the solution add dilute NaOH solution drop by drop till it is in excess.	White precipitate is obtained which dissolves in excess to produce colourless solution.	Zinc (II) ion (Zn^{2+}) confirmed.
To another part of solution add 1 mL of potassium ferrocyanide. Shake well.	A grey white precipitate is obtained.	Zinc (II) ion (Zn^{2+}) confirmed.
(B) **Manganese (II) ion (Mn^{2+})** Formation of buff coloured precipitate indicates the presence of Mn^{2+} ions.	Buff precipitate obtained	Manganese (II) ion (Mn^{2+}) may be present.
Dissolve the buff coloured precipitate into 1 ml of dilute HCl. Dilute it with 2–3 ml of water.	A clear solution is obtained.	
To this solution add dilute NaOH solution drop by drop till it is in excess.	A white precipitate is obtainedwhich turns brown due to aerial oxidation.	Manganese (Mn^{2+}) is confirmed.
(C) **Nickel (II) ion, (Ni^{2+})** Formation of black precipitate indicates the presence of Ni^{2+} ion.	Black precipitate is obtained.	Nickel (II) ion Ni^{2+} may be present.
Take a small amount of washed precipitate in a china dish. Add 1–2 mL of concentated HNO_3 to it. Boil it. Evaporate the contents of china dish to dryness.	A yellowish green residue is obtained.	

Cool down the china dish Add 2–3 mL of water. Shake well. Transfer this solution to a clean test tube.	A ligh green solution is obtained.	
Add excess of NH_4OH solution and then 1 mL of dimethyl glyoxime. Shake well.	A cherry red precipitate is obtained.	Nickel (II) ion (Ni^{2+}) confirmed.

(vi) **Work Sheet No. 6 :** Group V Calcium (ii) ion.

Experiment	Observation	Inference
Take 2 mL of original solution in a clean test tube. Add a pinch of solid NH_4Cl and excess of NH_4OH. solution and then 2 mL of freshly prepared $(NH_4)_2CO_3$ solution. shake well.	A white precipitate is obtained.	Calcium (II) ion (Ca^{2+}) may be present.
Formation of white precipitate indicates the presence of Ca^{2+} ion.		
Take a small amount of white precipitate in 1 mL of acetic acid.	A clear soln. is obtained.	
To the above obtained solution add 2 mL of ammonium oxalate solution. Shake well.	A white precipitate slowly appears.	Calcium (II) ion, (Ca^{2+}) confirmed.

An Illustration of identification of cation and anion in a given sample of salt

AIM : To find out the presence of an anion and a cation in the given salt

	Experiment	Observations	Inference
1.	Physical Examination (i)Colour of salt	white	May contain, Zn^{2+}, Pb^{2+}, Al^{3+}, Ca^{2+}, NH_4^+ may
	(ii)Heat small amount of salt in a clean dry test tube.	Salt sublimates	May contain NH_4^+ ions
(a)	**Anion analysis**		
2.	A pinch of salt was taken in a clean test tube 2 ml of dilute H_2SO_4 was added to it and heated first gently and then strongly.	No observable changes took place.	CO_3^{2-} S^{2-} and NO_2^- absent

3. A pinch of salt was taken in a clean and dry test tube. Added few drops of conc. H_2SO_4 to it, heated gently, few copper turning were added and contents were heated again.	A brown gas with pungent smell is given out and solution turns brown.	Nitrate ion (NO_3^-) ion may be present.
4. **Confirmation test of NO_3^- ion** : 2 ml of original soln. were taken in a test tube this 2 ml of freshly prepared $FeSO_4$ (aq) was added. Shaken well. To this added a few drops of concentrated H_2SO_4 along the side of test tube without disturbing the test tube.	A brown ring is formed at the junction, where the two liquids meet each other.	Nitrate (NO_3^-) ion confirmed.
5. Take a pinch of salt in a clean test tube 2 ml of concentrated NaOH solution added to it. warmed gently and then strongly.	No observable changes took place.	Ammonium ion (NH_4^+) absent.
(b) Cation Analysis		
6. A pinch of salt was taken in a test tube 2–3 ml water was added to it–followed by 3-4 drops of water HCl. Shake well.	A clear solution was obtained.	Lead (II) ion, (Pb^{2+}) absent.
7. To the above solution pass H_2S gas.	No precipitate obtained.	Group II is absent
8. 2 ml of original solution was taken in a test tube. Solid NH_4Cl was added in excess. followed by addition of excess of NH_4OH solution warm gently.	No precipitate obtained.	Group III is absent
9. Passed H_2S gas through group III solution.	No precipitate obtained.	Group IV absent
10. 2 ml of original soln. was taken in a test tube. A pinch of NH_4Cl solid was added. Excess of NH_4OH solution was added followed by addition of 2 ml of freshly prepared $(NH_4)_2CO_3$ solution. Shake well.	A white precipitate is obtained.	Group V may be present
Confirmation test of Cation		
11. Filtered and washed precipate was dissolved in 2 ml of acetic acid.	A clear soluion is obtained.	
12. 2 ml of $(NH_4)_2CO_3$ soln. was added to above obtained solution.	A white precipitate appeared slowly.	Calcium ion, (Ca^{2+}) confirmed.

Result : The given salt contains

Anion – NO_3^-

Cation – Ca^{2+}

Precautions

1. Use acids carefully.
2. Avoid using large quantities of acids and salt.
3. Keep the materials ready for testing the gas evolved.
4. Performed the test on the gas evolved immediately. If for any reason the testing is delayed, then add a little amount of more salt land test the gas immediately.
5. Addition of group reagents should be followed in correct order.
6. Analysis of anion should be carried out before cation analysis.
7. Pass H_2S gas through hot solution.
8. Always use a pinch of NH_4Cl in group V.

Observations

The students should record the observations and inference during the analysis of given salt (s).

14.7 CONCLUSION

The given salt contains

(i) Acid radical.....

(ii) Basic radical.....

The given salt is.....

14.8 CHECK YOUR UNDERSTAING

1. *What is meant by qualitative analysis ?*

   .....

2. *What is meant by the terms positive test and negative test ?*

   .....

3. *What happens when carbon dioxide is passed through $Ba(OH)_2$ solution instead of calcium hydroxide solution ?*

   .....

4. *What happens when an acid is over boiled ?*

   .....

5. *Why is it necessary to keep the test tube dry during chromyl chloride test ?*

   .....

6. *Why Bromine (Br_2) / Iodine (I_2) (g) is more soluble in organic solvent such as CCl_4 than in water?*

.....

7. *How will you distinguish between Br and NO_3 in qualitative analysis ?*

.....

8. *Why can HNO_3 not be used to prepare original solution of a salt ?*

.....

9. *What is a group reagent ?*

.....

10. *What chemicals are used in kipps apparatus to make hydrogen sulphide gas, (H_2S)?*

.....

11. *What is the group V reagent ?*

.....

12. *Why is it necessary to add concentrated H_2SO_4 along the sides of the test tube during ring test of NO_3 ion ?*

.....

13. *Can group V precipitate dissolve in dilute HCl instead of acetic acid ?*

.....

14.9 NOTE FOR THE TEACHER

1. Teacher should explain important terms used during experiment.
2. Teacher should demonstrate all the techniques explained in the experiment.
3. In qualitative analysis, students use concentrated acids, alkali and other laboratory reagents. The teacher should be cautions and ensure that all students follow proper precautions
4. Freshly prepared soln. of (a) $FeSO_4$ (b) sodium nitroprusside (c) $(NH_4)_2CO_3$ should be used.
5. Following combination of anions and cations should not be given

(i) Halide ions and Pb^{2+}
(ii)SO_4^{2-} and Pb^{2+}, Pb^{2+}, Ca^{2+}
(iii)PO_4^{3-} and gp III onwards
(iv)NO_3^- and gp II,
(v)S^{2-} and group II and group IV cations

6. The teacher may ensure that the students carry out the analysis of given salts systematically.
7. Follow all the precautions given in this experiments and general precautions of a chemistry laboratory.
8. Get sufficient practice of analysis of salts. The students may use their note-books to records the practicals.

14.10 Check your Answers

Ans.1. Qualitative analysis is the scheme of the detection and identification of the constituent ions of an unknown salt.

Ans.2. A positive test is the one which gives the observations, needed for that radical. A negative test does not give the required observations for a test.

Ans.3. $Ba(OH)_2 + CO_2 \longrightarrow BaCO_3 + H_2O$

Ans.4. $2H_2SO_4 \longrightarrow 2H_2O + 2SO_2 + O_2$

Ans.5. If dry test tube is not taken. Concentrated H_2SO_4 gets diluted and chromyl chloride test can not be performed properly.

Ans.6. Being covalent in nature Br_2 or I_2 are more soluble in non–polar solvent such as CCl_4

Ans.7. Bromide ions, (Br^-) gives reddish brown vapour of Br_2 and solution turns red in colours on treatment with concentrated H_2SO_4 NO_3^- ion gives brown fumes of NO_2 which intensified on heating with copper turning.

Ans.8. HNO_3 is an oxidising agent. It oxidises H_2S to S (sulphur).

Ans.9. The cations in their respective group are precipitated by adding certain chemicals under definite condition of acidic or alkanity. These chemicals are called group reagents.

Ans.10. FeS and dilute H_2SO_4

Ans.11. Group V reagent $(NH_4)_2CO_3$ solution in the presence of NH_4Cl and NH_4OH

Ans.12. If the concentrated H_2SO_4 is added directly, while solution turn reddish brown.

Ans.13. Ca^{2+} ion is precipilated as calcium oxalate, ($Ca C_2O_4$) on adding ammonium oxalate $(NH_4)_2 C_2O_4$. This precipitate is highly soluble in dilute HCl. Hence calcium (II) ion will not precipitate in presence of dilute HCl.