SALT ANALYSIS

Analysis always does not mean breaking of substance into its ultimate constituents. Finding out the nature of substance and identity of its constituents is also analysis and is known as ***qualitative analysis.*** Qualitative analysis of inorganic salts means the identification of cations and anions present in the salt or a mixture of salts. Inorganic salts may be obtained by complete or partial neutralisation of acid with base or vice-versa. In the formation of a salt, the part contributed by the ***acid*** is called ***anion*** and the part contributed by the ***base*** is called ***cation***. For example, in the salts $CuSO_4$ and NaCl, Cu^{2+} and Na^+ ions are cations and SO_4^{2-} and Cl^- ions are anions. Qualitative analysis is carried out on various scales. Amount of substance employed in these is different. In macro analysis, 0.1 to 0.5 g of substance and about 20 mL of solution is used. For semimicro analysis, 0.05 g substance and 1 mL solution is needed while for micro analysis amount required is very small. Qualitative analysis is carried out through the reactions which are easily perceptible to our senses such as sight and smell. Such reactions involve:

(a) Formation of a precipitate
(b) Change in colour
(c) Evolution of gas etc.

Systematic analysis of an inorganic salt involves the following steps:

(i) Preliminary examination of solid salt and its solution.
(ii) Determination of anions by reactions carried out in solution (wet tests) and confirmatory tests.
(iii) Determination of cations by reactions carried out in solution (wet tests) and confirmatory tests.

Although these tests are not conclusive but sometimes they give quite important clues for the presence of certain anions or cations. These tests can be performed within 10^{-15} minutes. These involve noting the general appearance and physical properties, such as colour, smell, solubility etc. of the salt. These are named as ***dry tests.***

Heating of dry salt, blow pipe test, flame tests, borax bead test, sodium carbonate bead test, charcoal cavity test etc. come under dry tests.

Solubility of a salt in water and the pH of aqueous solutions give important information about the nature of ions present in the salt. If a solution of the salt is acidic or basic in nature, this means that it is being hydrolysed in water. If the solution is basic in nature then salt may be some carbonate or sulphide etc. If the solution shows acidic nature then it may be an acid salt or salt of weak base and strong acid. In this case it is best to neutralise the solution with sodium carbonate before testing it for anions.

Gases evolved in the preliminary tests with dil. H_2SO_4/dil. HCl and conc. H_2SO_4 also give good indication about the presence of acid radicals (See Tables 1 and 3). ***Preliminary tests should always be performed before starting the confirmatory tests for the ions.***

EXPERIMENT 1.1

Aim

To detect one cation and one anion in the given salt from the following ions:

Cations - Pb^{2+}, Cu^{2+}, As^{3+}, Al^{3+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, Zn^{2+}, Co^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+},NH_4^+

Anions - CO_3^{2-} , S^{2-}, SO_4^{2-}, NO_2^- , NO_3^-, Cl^-, Br^-, I^-, PO_4^{3-}, CH_3COO^-.

(Insoluble salts to be excluded)

Theory

Two basic principles of great use in the analysis are:

(i) the Solubility product

(ii) the Common ion effect.

When ionic product of a salt exceeds its solubility product, precipitation takes place. Ionic product of salt is controlled by making use of common ion effect.

Material Required

• Boiling tube	:	As per need
• Test tubes	:	As per requirement
• Measuring cylinder	:	One
• Test tube stand	:	One
• Test tube holder	:	One
• Delivery tube	:	One
• Corks	:	As per need
• Filter paper	:	As per need
• Reagents	:	As per need

Step - I : Preliminary Test with Dilute Sulphuric Acid → In this test the action of dilute sulphuric acid (procedure is given below) on the salt is noted at room temperature and on warming.

Carbonate (CO_3^{2-}), sulphide (S^{2-}), sulphite (SO_3^{2-}), nitrite (NO_2^-) and acetate (CH_3COO^-) react with dilute sulphuric acid to evolve different gases. Study of the characteristics of the gases evolved gives information about the anions. Summary of characteristic properties of gases is given in Table 1.

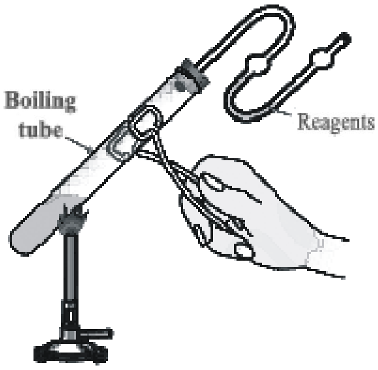


Fig. 1.1 Testing a Gas

Procedure

(a) Take 0.1 g of the salt in a test tube and add 1–2 mL of dilute sulphuric acid. Observe the change, if any, at room temperature. If no gas is evolved, warm the content of the test tube. If gas is evolved test it by using the apparatus shown in Fig.1 and identify the gas evolved (See Table 1).

Table 1 : Preliminary test with dilute sulphuric acid

Observations	Inference	
	Gas Evolved	Possible Anion
A colourless, odourless gas is evolved with brisk effervescence, which turns lime water milky.	CO_2	Carbonate (CO_3^{2-})
Colourless gas with the smell of rotten eggs is evolved which turns lead acetate paper black.	H_2S	Sulphide (S^{2-})
Colourless gas with a pungent smell, like burning sulphur which turns acidified potassium dichromate solution green.	SO_2	Sulphite (SO_3^{2-})
Brown fumes which turn acidified potassium iodide solution containing starch solution blue.	NO_2	Nitrite (NO_2^-)
Colourless vapours with smell of vinegar. Vapours turn blue litmus red.	CH_3COOH vapours	Acetate, (CH_3COO^-)

Confirmatory tests for CO_3^{2-} , S^{2-}, SO_3^{2-}, NO_2^- and CH_3COO^-

Confirmatory (wet) tests for anions are performed by using water extract when salt is soluble in water and by using sodium carbonate extract when salt is insoluble in water. Confirmation of CO_3^{2-} is done by using aqueous solution of the salt or by using solid salt as such because sodium carbonate extract contains carbonate ions. Water extract is made by dissolving salt in water. Preparation of sodium carbonate extract is given below.

Preparation of sodium carbonate extract

Take 1 g of salt in a porcelain dish or boiling tube. Mix about 3 g of solid sodium carbonate and add 15 mL of distilled water to it. Stir and boil the content for about 10 minutes. Cool, filter and collect the filtrate in a test tube and label it as sodium carbonate extract.

Confirmatory tests for acid radicals, which react with dilute sulphuric acid are given in Table 2.

Table 2 : Confirmatory tests for CO_3^{2-}, S^{2-}, SO_3^{2-}, NO_3^-, CH_3COO^-

Anion	Confirmatory Test
Carbonate (CO_3^{2-})	Take 0.1 g of salt in a test tube, add dilute sulphuric acid. CO_2 gas is evolved with brisk effervescence which turns lime water milky. On passing the gas for some more time, milkiness disappears.
Sulphide (S^{2-})	Take 1 mL of water extract and make it alkaline by adding ammonium hydroxide or sodium carbonate extract. Add a drop of sodium nitroprusside solution. Purple or violet colouration appears.
Sulphite (SO_3^{2-})	(a) Take 1 mL of water extract or sodium carbonate extract in a test tube and add barium chloride solution. A white precipitate is formed which dissolves in dilute hydrochloric acid and sulphur dioxide gas is also evolved (b) Take the precipitate of step (a) in a test tube and add a few drops of potassium permanganate solution acidified with dil. H_2SO_4. Colour of potassium permanganate solution gets discharged.
Nitrite (NO_2^-)	(a) Take 1 mL of water extract in a test tube. Add a few drops of potassium iodide solution and a few drops of starch solution, acidify with acetic acid. Blue colour appears. (b) Acidify 1 mL of water extract with acetic acid. Add 2-3 drops of sulphanilic acid solution followed by 2-3 drops of 1-naphthylamine reagent. Appearance of red colour indicates the presence of nitrite ion.
Acetate, (CH_3COO^-)	(a) Take 0.1 g of salt in a china dish. Add 1 mL of ethanol and 0.2 mL conc. H_2SO_4 and heat. Fruity odour confirms the presence of acetate ion. (b) Take 0.1 g of salt in a test tube, add 1-2 mL distilled water, shake well filter if necessary. Add 1 to 2 mL neutral ferric chloride solution to the filtrate. Deep red colour appears which disappears on boiling and a brown-red precipitate is formed.

Chemistry of Confirmatory Tests

1. Test for Carbonate ion [CO_3^{2-}]

If there is effervescence with the evolution of a colourless and odourless gas on adding dil. H_2SO_4 to the solid salt, this indicates the presence of carbonate ion.

The gas turns lime water milky due to the formation of $CaCO_3$

$$Na_2CO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + CO_2$$
$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 + H_2O$$

If CO_2 gas is passed in excess through lime water, the milkiness disappears due to the formation of calcium hydrogen carbonate which is soluble in water.

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

2. Test for Sulphide ion [S^{2-}]

(a) With warm dilute H_2SO_4 a sulphide gives hydrogen sulphide gas which smells like rotten eggs. A piece of filter paper dipped in lead acetate solution turns black on exposure to the gas due to the formation of lead sulphide which is black in colour.

$Na_2S + H_2SO_4 \longrightarrow Na_2SO_4 + H_2S$

$(CH_3COO)_2Pb + H_2S \longrightarrow PbS + 2CH_3COOH$

Lead sulphide

Black precipitate

(b) If the salt is soluble in water, take the solution of salt in water make it alkaline with ammonium hydroxide and add sodium nitroprusside solution. If it is insoluble in water take sodium carbonate extract and add a few drops of sodium nitroprusside solution. Purple or violet colouration due to the formation of complex compound $Na_4[Fe(CN)_5NOS]$ confirms the presence of sulphide ion in the salt.

$Na_2S + Na_2[Fe(CN)_5NO] \longrightarrow Na_4[Fe(CN)_5NOS]$

Sodium nitroprusside Complex of Purple colour

3. Test for Sulphite ion [SO_3^{2-}]

(a) On treating sulphite with warm dil. H_2SO_4, SO_2 gas is evolved which is suffocating with the smell of burning sulphur.

$Na_2SO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + SO_2$

The gas turns potassium dichromate paper acidified with dil. H_2SO_4, green.

$K_2Cr_2O_7 + H_2SO_4 + 3SO_2 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + H_2O$

Chromium

sulphate (green)

(b) An aqueous solution or sodium carbonate extract of the salt produces a white precipitate of barium sulphite on addition of barium chloride solution.

$Na_2SO_3 + BaCl_2 \longrightarrow 2NaCl + BaSO_3$

White ppt.

This precipitate gives following tests.

(i) This precipitate on treatment with dilute HCl, dissolves due to decomposition of sulphite by dilute HCl. Evolved SO_2 gas can be tested.

$BaSO_3 + 2HCl \longrightarrow BaCl_2 + H_2O + SO_2$

(ii) Precipitate of sulphite decolourises acidified potassium permanganate solution.

$BaSO_3 + H_2SO_4 \longrightarrow BaSO_4 + H_2O + SO_2$

$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5\,[O]$

$SO_2 + H_2O + [O] \longrightarrow H_2SO_4$

4. Test for Nitrite ion [NO_2^-]

(a) On treating a solid nitrite with dil. H_2SO_4 and warming , reddish brown fumes of NO_2 gas are evolved. Addition of potassium iodide solution to the salt solution followed by freshly prepared starch solution and acidification with acetic acid produces blue colour. Alternatively, a filter paper moistened with potassium iodide and starch solution and a few drops of acetic acid turns blue on exposure to the gas, due to the interaction of liberated iodine with starch.

(i) $2NaNO_2 + H_2SO_4 \longrightarrow Na_2SO_4 + 2HNO_2$

$3HNO_2 \longrightarrow HNO_3 + 2NO + H_2O$ (disproportionation)

$2NO + O_2 \longrightarrow 2NO_2$

Brown gas

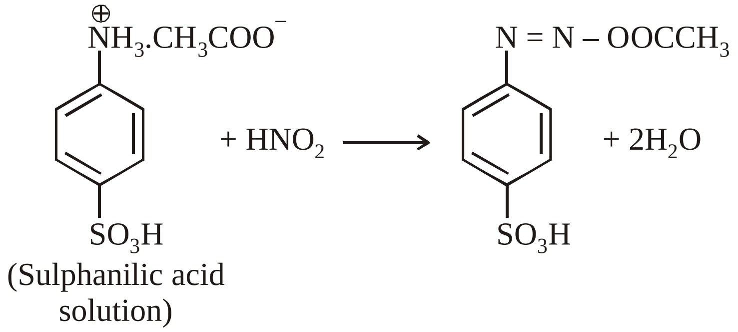
(ii) $NO_2^- + CH_3COOH \longrightarrow HNO_2 + CH_3COO^-$

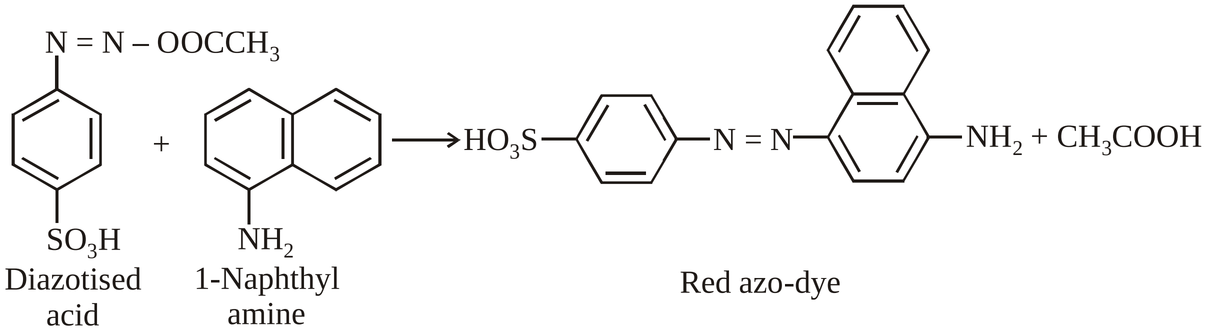
$2HNO_2 + 2KI + 2CH_3COOH \longrightarrow 2CH_3COOK + 2H_2O + 2NO + I_2$

$I_2 + \text{Starch} \longrightarrow \text{Blue complex}$

(b) Sulphanilic acid — 1-naphthylamine reagent test (Griess-llosvay test) : On adding sulphanilic acid and 1-naphthylamine reagent to the water extract or acidified with acetic acid, sulphanilic acid is diazotised in the reaction by nitrous acid formed. Diazotised acid couples with 1-naphthylamine to form a red azo-dye.

$$NO_2^- + CH_3COOH \rightarrow HNO_2 + CH_3COO^-$$





The test solution should be very dilute. In concentrated solutions reaction does not proceed beyond diazotisation.

5. **Test for Acetate ion [CH_3COO^-]**

(a) If the salt smells like vinegar on treatment with dil. H_2SO_4, this indicates the presence of acetate ions. Take 0.1 g of salt in a china dish and add 1 mL of ethanol. Then add about 0.2 mL of conc. H_2SO_4 and heat. Fruity odour of ethyl acetate indicates the presence of CH_3COO^- ion.

$$2CH_3COONa + H_2SO_4 \longrightarrow Na_2SO_4 + 2CH_3COOH$$

$$CH_3COOH + C_2H_5OH \longrightarrow \underset{\text{(Fruity odour)}}{\underset{\text{Ethylacetate}}{CH_3COOC_2H_5}} + H_2O$$

(b) Acetate gives deep red colour on reaction with neutral ferric chloride solution due to the formation of complex ion which decomposes on heating to give Iron (III) dihydroxyacetate as brown red precipitate.

$$6CH_3COO^- + 3Fe^{3+} + 2H_2O \longrightarrow [Fe_3(OH)_2(CH_3COO)_6]^+ + 2H^+$$

$$[Fe_3(OH)_2(CH_3COO)_6]^+ + 4H_2O \longrightarrow \underset{\text{(Brown-red precipitate)}}{\underset{\text{Iron(III)dihydroxyacetate}}{3[Fe(OH)_2(CH_3COO)]}} + 3CH_3COOH + H^+$$

Step-II : Preliminary Test with Concentrated Sulphuric Acid If no positive result is obtained from dil. H_2SO_4 test, take 0.1 g of salt in a test tube and 3-4 drops of conc. H_2SO_4. Observe the change in the reaction mixture in cold and then warm it. Identify the gas evolved on heating (see Table 3).

Table 3 : Preliminary examination with concentrated sulphuric acid

Observations	Inference	
	Gas/Vapours Evolved	**Possible Anion**
A colourless gas with pungent smell, which gives dense white fumes when a rod dipped in ammonium hydroxide is brought near the mouth of the test tube.	HCl	Chloride, (Cl^-)
Reddish brown gas with a pungent odour is evolved. Intensity of reddish gas increases on heating the reaction mixture after addition of solid MnO_2 to the reaction mixture. Solution also acquires red colour.	Br_2 vapours	Bromide, (Br^-)
Violet vapours, which turn starch paper blue and a layer of violet sublimate is formed on the sides of the tube. Fumes become dense on adding MnO_2 to the reaction mixture.	I_2 vapours	Iodide, (I^-)
Brown fumes evolve which become dense upon heating the reaction mixture after addition of copper turnings and the solution acquires blue colour.	NO_2	Nitrate, (NO_3^-)
Colourless, odourless gas is evolved which turns lime water milky and the gas coming out of lime water burns with a blue flame, if ignited.	CO and CO_2	Oxalate, ($C_2O_4^{2-}$)

Table 4 : Confirmatory tests for Cl^-, Br^-, I^-, NO_3^- and $C_2O_4^{2-}$

Anion	**Confirmatory Test**
Chloride (Cl^-)	(a) Take 0.1 g of salt in a test tube, add a pinch of manganese dioxide and 3-4 drops of conc. sulphuric acid. Heat the reaction mixture. Greenish yellow chlorine gas is evolved which is detected by its pungent odour and bleaching action. (b) Take 1 mL of sodium carbonate extract in a test tube, acidify it with dil. HNO_3 or take water extract and add silver nitrate solution. A curdy white precipitate is obtained which is soluble in ammonium hydroxide solution. (c) Take 0.1 g salt and a pinch of solid potassium dichromate in a test tube, add conc. H_2SO_4, heat and pass the gas evolved through sodium hydroxide solution. It becomes yellow. Divide the solution into two parts. Acidify one part with acetic acid and add lead acetate solution. A yellow precipitate is formed. Acidify the second part with dilute sulphuric acid and add 1 mL of amyl alcohol followed by 1 mL of 10% hydrogen peroxide. After gentle shaking the organic layer turns blue.
Bromide (Br^-)	(a) Take 0.1 g of salt and a pinch of MnO_2 in a test tube. Add 3-4 drops conc.sulphuric acid and heat. Intense brown fumes are evolved. (b) Neutralise 1 mL of sodium carbonate extract with hydrochloric acid (or take the water extract). Add 1 mL carbon tetrachloride (CCl_4)/ chloroform ($CHCl_3$)/ carbon disulphide. Now add an excess of chlorine water dropwise and shake the test tube. A brown colouration in the organic layer confirms the presence of bromide ion. (c) Acidify 1 mL of sodium carbonate extract with dil. HNO_3 (or take 1 mL water extract) and add silver nitrate solution. A pale yellow precipitate soluble with difficulty in ammonium hydroxide solution is obtained.
Iodide (I^-)	(a) Take 1 mL of salt solution neutralised with HCl and add 1 mL chloroform/carbon tetrachloride/carbon disulphide. Now add an excess of chlorine water drop wise and shake the test tube. A violet colour appears in the organic layer. (b) Take 1 mL of sodium carbonate extract acidify it with dil. HNO_3 (or take water extract). Add, silver nitrate solution. A yellow precipitate insoluble in NH_4OH solution is obtained.
*Nitrate (NO_3^-)	Take 1 mL of salt solution in water in a test tube. Add 2 mL of conc. H_2SO_4 and mix thoroughly. Cool the mixture under the tap. Add freshly prepared ferrous sulphate along the sides of the test tube without shaking. A dark brown ring is formed at the junction of the two solutions.
Oxalate ($C_2O_4^{2-}$)	(a) Take 1 mL of water extract or sodium carbonate extract acidified with acetic acid and add calcium chloride solution. A white precipitate insoluble in ammonium oxalate and oxalic acid solution but soluble in dilute hydrochloric acid and dilute nitric acid is formed. (b) Take the precipitate from test (a) and dissolve it in dilute H_2SO_4. Add very dilute solution of $KMnO_4$ and warm. Colour of KMnO solution is discharged. Pass the gas coming out through lime water. The lime water turns milky.

Chemistry of Confirmatory Tests

1. Test for Chloride ion [Cl^-]

(a) If on treatment with warm conc. H_2SO_4 the salt gives a colourless gas with pungent smell or if the gas which gives dense white fumes with ammonia solution, then the salt may contain Cl^- ions and the following reaction occurs.

$$NaCl + H_2SO_4 \longrightarrow \underset{\substack{\text{Sodium}\\\text{hydrogen sulphate}}}{NaHSO_4} + \underset{\substack{\text{Hydrogen chloride}\\\text{Colourless gas}}}{HCl}$$

$$HCl + NH_3 \longrightarrow \underset{\substack{\text{Ammonium chloride}\\\text{White fumes}}}{NH_4Cl}$$

(b) If a salt gives effervescence on heating with conc. H_2SO_4 and MnO_2 and a light greenish yellow pungent gas is evolved, this indicates the presence of Cl^-ions.

$$MnO_2 + 2NaCl + 2H_2SO_4 \longrightarrow Na_2SO_4 + MnSO_4 + 2H_2O + Cl_2$$

(c) Salt solution acidified with dilute HNO_3 on addition of silver nitrate solution gives a curdy white precipitate soluble in ammonium hydroxide solution. This indicates the presence of Cl^- ions in the salt.

$$NaCl + AgNO_3 \longrightarrow NaNO_3 + \underset{\substack{\text{Silver chloride}\\\text{(White precipitate)}}}{AgCl}$$

$$AgCl + 2NH_4OH \longrightarrow \underset{\substack{\text{Diammine silver (I)}\\\text{chloride}}}{[Ag(NH_3)_2]Cl} + 2H_2O$$

(d) Mix a little amount of salt and an equal amount of solid potassium dichromate ($K_2Cr_2O_7$) in a test tube and add conc. H_2SO_4 to it. Heat the test tube and pass the evolved gas through sodium hydroxide solution. If a yellow solution is obtained, divide the solution into two parts. Acidify the first part with acetic acid and then add lead acetate solution. Formation of a yellow precipitate of lead chromate confirms the presence of chloride ions in the salt. This test is called ***chromyl chloride test.***

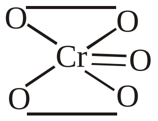
$$4NaCl + K_2Cr_2O_7 + 6H_2SO_4 \longrightarrow 2KHSO_4 + \underset{\substack{\text{(Chromyl}\\\text{chloride)}}}{2CrO_2Cl_2} + 4NaHSO_4 + 3H_2O$$

$$CrO_2Cl_2 + 4NaOH \longrightarrow Na_2CrO_4 + 2NaCl + 2H_2O$$

$$(CH_3COO)_2Pb + \underset{\substack{\text{Sodium}\\\text{chromate}}}{Na_2CrO_4} \longrightarrow \underset{\substack{\text{Lead chromate}\\\text{(Yellow precipitate)}}}{PbCrO_4} + 2CH_3COONa$$

Acidify the second part with dilute sulphuric acid and add small amounts of amyl alcohol and then 1 mL of 10% hydrogen peroxide solution. On gentle shaking organic layer turns blue. CrO_4^{2-} ion formed in the reaction of chromyl chloride with sodium hydroxide reacts with hydrogen peroxide to form chromium pentoxide (CrO_5) (See structure) which dissolves in amyl alcohol to give blue colour.

$$CrO_4^{2-} + 2H^+ + 2H_2O_2 \longrightarrow \underset{\substack{\text{Chromium}\\\text{pentoxide}}}{CrO_5} + 3H_2O$$



Structure of chromium pentoxide

2. Test for Bromide ion (Br^-)

If on heating the salt with conc. H_2SO_4 reddish brown fumes of bromine are evolved in excess, this indicates the presence of Br^- ions. The fumes get intensified on addition of MnO_2. Bromine vapours turn starch paper yellow.

$$2NaBr + 2H_2SO_4 \longrightarrow Br_2 + SO_2 + Na_2SO_4 + 2H_2O$$
$$2NaBr + MnO_2 + 2H_2SO_4 \longrightarrow Na_2SO_4 + MnSO_4 + 2H_2O + Br_2$$

(a) Add 1 mL of carbon tetrachloride (CCl_4)/chloroform ($CHCl_3$) and excess of freshly prepared chlorine water dropwise to the salt solution in water or sodium carbonate extract neutralised with dilute HCl. Shake the test tube vigorously. The appearance of an orange brown colouration in the organic layer due to the dissolution of bromine in it, confirms the presence of bromide ions.

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

(b) Acidify the sodium carbonate extract of the salt with dil. HNO_3. Add silver nitrate ($AgNO_3$) solution and shake the test tube. A pale yellow precipitate is obtained which dissolves in ammonium hydroxide with difficulty.

$$NaBr + AgNO_3 \longrightarrow NaNO_3 + \underset{\substack{\text{Silver bromide} \\ \text{Pale yellow precipitate}}}{AgBr}$$

3. Test for Iodide ion (I^-)

(a) If on heating the salt with conc. H_2SO_4, deep violet vapours with a pungent smell are evolved. These turns starch paper blue and a violet sublimate is formed on the sides of the test tube, it indicates the presence of I^- ions. Some HI, sulphur dioxide, hydrogen sulphide, and sulphur are also formed due to the following reactions.

$$2NaI + 2H_2SO_4 \longrightarrow Na_2SO_4 + SO_2 + 2H_2O + I_2$$
$$I_2 + \underset{\text{solution}}{\text{Starch}} \longrightarrow \text{Blue colour}$$
$$NaI + H_2SO_4 \longrightarrow NaHSO_4 + HI$$
$$2HI + H_2SO_4 \longrightarrow 2H_2O + I_2 + SO_2$$
$$6NaI + 4H_2SO_4 \longrightarrow 3I_2 + 4H_2O + S + 3Na_2SO_4$$
$$8NaI + 5H_2SO_4 \longrightarrow 4I_2 + H_2S + 4Na_2SO_4 + 4H_2O$$

On adding MnO_2 to the reaction mixture, the violet vapours become dense.

$$2NaI + MnO_2 + 2H_2SO_4 \longrightarrow I_2 + MnSO_4 + Na_2SO_4 + 2H_2O$$

(b) Add 1 mL of $CHCl_3$ or CCl_4 and chlorine water in excess to the salt solution in water or sodium carbonate extract neutralised with dil.HCl and shake the test tube vigorously. Presence of violet colouration in the organic layer confirms the presence of iodide ions.

$$2NaI + Cl_2 \longrightarrow 2NaCl + I_2$$

Iodine dissolves in the organic solvent and the solution becomes violet.

(c) Acidify sodium carbonate extract of the salt with dil.HNO_3 and add $AgNO_3$ solution. Appearance of a yellow precipitate insoluble in excess of NH_4OH confirms the presence of iodide ions.

$$NaI + AgNO_3 \longrightarrow \underset{\substack{\text{silver iodide} \\ \text{(Yellow precipitate)}}}{AgI} + NaNO_3$$

(a) If on heating the salt with conc. H_2SO_4 light brown fumes are evolved then heat a small quantity of the given salt with few copper turnings or chips and conc. H_2SO_4. Evolution of excess of brown fumes indicates the presence of nitrate ions. The solution turns blue due to the formation of copper sulphate.

$$NaNO_3 + H_2SO_4 \longrightarrow NaHSO_4 + HNO_3$$

$$4HNO_3 \longrightarrow 4NO_2 + O_2 + 2H_2O$$

$$2NaNO_3 + 4H_2SO_4 + 3Cu \longrightarrow \underset{\substack{\text{Copper sulphate}\\ \text{(Blue)}}}{3CuSO_4} + Na_2SO_4 + 4H_2O + 2NO$$

$$2NO + O_2 \longrightarrow \underset{\text{(Brown fumes)}}{2NO_2}$$

(b) Take 1 mL of an aqueous solution of the salt and add 2 mL conc. H_2SO_4 slowly. Mix the solutions thoroughly and cool the test tube under the tap. Now, add freshly prepared ferrous sulphate solution along the sides of the test tube dropwise so that it forms a layer on the top of the liquid already present in the test tube. A dark brown ring is formed at the junction of the two solutions due to the formation of nitroso ferrous sulphate (Fig. 1.2). Alternatively first ferrous sulphate is added and then concentrated sulphuric acid is added.

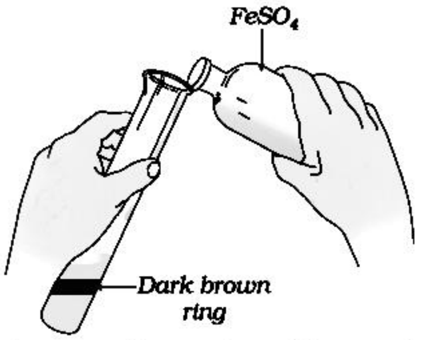


Fig. 1.2 : *Formation of brown ring*

$$NaNO_3 + H_2SO_4 \longrightarrow NaHSO_4 + HNO_3$$

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

$$FeSO_4 + NO \longrightarrow \underset{\substack{\text{Nitroso ferrous sulphate}\\ \text{(Brown)}}}{[Fe(NO)]SO_4}$$

5. Test for Oxalate ion [$C_2O_4^{2-}$]

If carbon dioxide gas along with carbon monoxide gas is evolved in the preliminary examination with concentrated sulphuric acid, this gives indication about the presence of oxalate ion.

$$Na_2C_2O_4 + \text{Conc.}H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + CO_2\uparrow + CO\uparrow$$

Oxalate is confirmed by the following tests:

(a) Acidify sodium carbonate extract with acetic acid and add calcium chloride solution. A white precipitate of calcium oxalate, insoluble in ammonium oxalate and oxalic acid solution indicates the presence of oxalate ion.

$$CaCl_2 + Na_2C_2O_4 \longrightarrow \underset{\substack{\text{Calcium oxalate}\\ \text{(White precipitate)}}}{CaC_2O_4} + 2NaCl$$

(b) $KMnO_4$ test

Filter the precipitate from test (a). Add dil. H_2SO_4 to it followed by dilute $KMnO_4$ solution and warm. Pink colour of $KMnO_4$ is discharged:

$$CaC_2O_4 + H_2SO_4 \longrightarrow \underset{\text{Calcium sulphate}}{CaSO_4} + \underset{\text{Oxalic acid}}{H_2C_2O_4}$$

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \longrightarrow 2MnSO_4 + K_2SO_4 + 8H_2O + 10CO_2$$

Pass the gas evolved through lime water. A white precipitate is formed which dissolves on passing the gas for some more time.

Step-III : Test for Sulphate and Phosphate

If no positive test is obtained in Steps-I and II, then tests for the presence of sulphate and phosphate ions are performed. These tests are summarised in Table 5.

Table 5 : Confirmatory tests for Sulphate and Phosphate

Ion	Confirmatory Test
Sulphate (SO_4^{2-})	(a) Take 1 mL water extract of the salt in water or sodium carbonate and after acidifying with dilute hydrochloric acid add $BaCl_2$ solution. White precipitate insoluble in conc. HCl or conc. HNO_3 is obtained. (b) Acidify the aqueous solution or sodium carbonate extract with acetic acid and add lead acetate solution. Appearance of white precipitate confirms the presence of SO_4^{2-} ion.
Phosphate (PO_4^{3-})	(a) Acidify sodium carbonate extract or the solution of the salt in water with conc. HNO_3 and add ammonium molybdate solution and heat to boiling. A canary yellow precipitate is formed.

1. **Test of Sulphate ions [SO_4^{2-}]**

(a) Aqueous solution or sodium carbonate extract of the salt acidified with acetic acid on addition of barium chloride gives a white precipitate of barium sulphate insoluble in conc. HCl or conc. HNO_3.

$$Na_2SO_4 + BaCl_2 \longrightarrow \underset{\substack{\text{Barium sulphate}\\ \text{(White precipitate)}}}{BaSO_4} + 2NaCl$$

(b) Sulphate ions give white precipitate of lead sulphate when aqueous solution or sodium carbonate extract neutralised with acetic acid is treated with lead acetate solution.

$$Na_2SO_4 + (CH_3COO)_2Pb \longrightarrow \underset{\substack{\text{Lead sulphate}\\ \text{(White precipitate)}}}{PbSO_4} + 2CH_3COONa$$

2. **Test for Phosphate ion [PO_4^{3-}]**

(a) Add conc. HNO_3 and ammonium molybdate solution to the test solution containing phosphate ions and boil. A yellow colouration in solution or a canary yellow precipitate of ammonium-phosphomolybdate, $(NH_4)_3[P(Mo_3O_{10})_4]$ is formed. Each oxygen of phosphate has been replaced by Mo_3O_{10} group.

$$Na_2HPO_4 + 12(NH_4)_2MoO_4 + 23HNO_3 \longrightarrow \underset{\substack{\text{Canary yellow}\\ \text{precipitate}}}{(NH_4)_3[P(Mo_3O_{10})_4]} + 2NaNO_3 + 21NH_4NO_3 + 12H_2O$$

HEATING DEVICES

Heating during the laboratory work can be done with the help of a gas burner, spirit lamp or a kerosene lamp. The gas burner used in the laboratory is usually Bunsen burner . Various parts of Bunsen burner are shown in Fig. The description of these parts is as follows :

(A) Parts of Bunsen Burner

1. The Base

Heavy metallic base is connected to a side tube called gas tube. Gas from the source enters the burner through the gas tube and passes through a small hole called Nipple or Nozzle and enters into the burner tube under increased pressure and can be burnt at the upper end of the burner tube.

2. The Burner Tube

It is a long metallic tube having two holes diametrically opposite to each other near the lower end which form the air vent. The tube can be screwed at the base. The gas coming from the nozzle mixes with the air coming through the air vent and burns at its upper end.

3. The Air Regulator

It is a short metallic cylindrical sleeve with two holes diametrically opposite to each other. When it is fitted to the burner tube, it surrounds the air vent of the burner tube. To control the flow of air through the air vent, size of its hole is adjusted by rotating the sleeve.

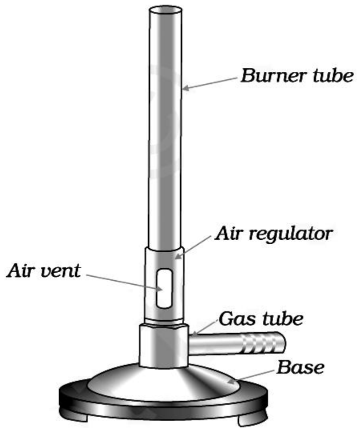


Fig. Bunsen burner

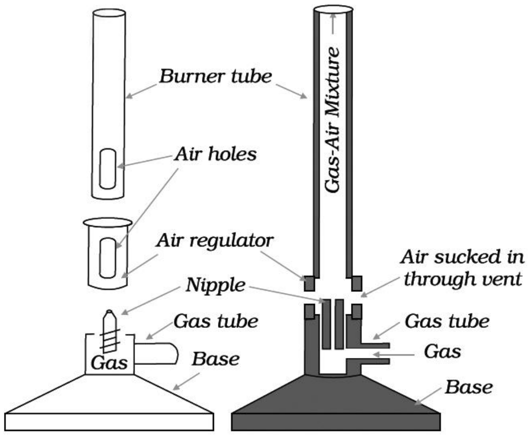


Fig. Parts of Bunsen barner

If the air vent is closed and the gas is ignited, the flame will be large and luminous (smoky and yellow in colour). The light emitted by the flame is due to the radiations given off by the hot carbon particles of partially burnt fuel. The temperature of the flame in this situation is low. If adjustment of sleeve on vent is such that gas mixed with air is fed into the flame, the flame becomes less luminous and finally turns blue. When the flow of air is correctly adjusted, the temperature of the flame becomes quite high. This is called non-luminous flame. Various zones of flame are shown below in Fig.

Three distinctly visible parts of the Bunsen flame are described below :

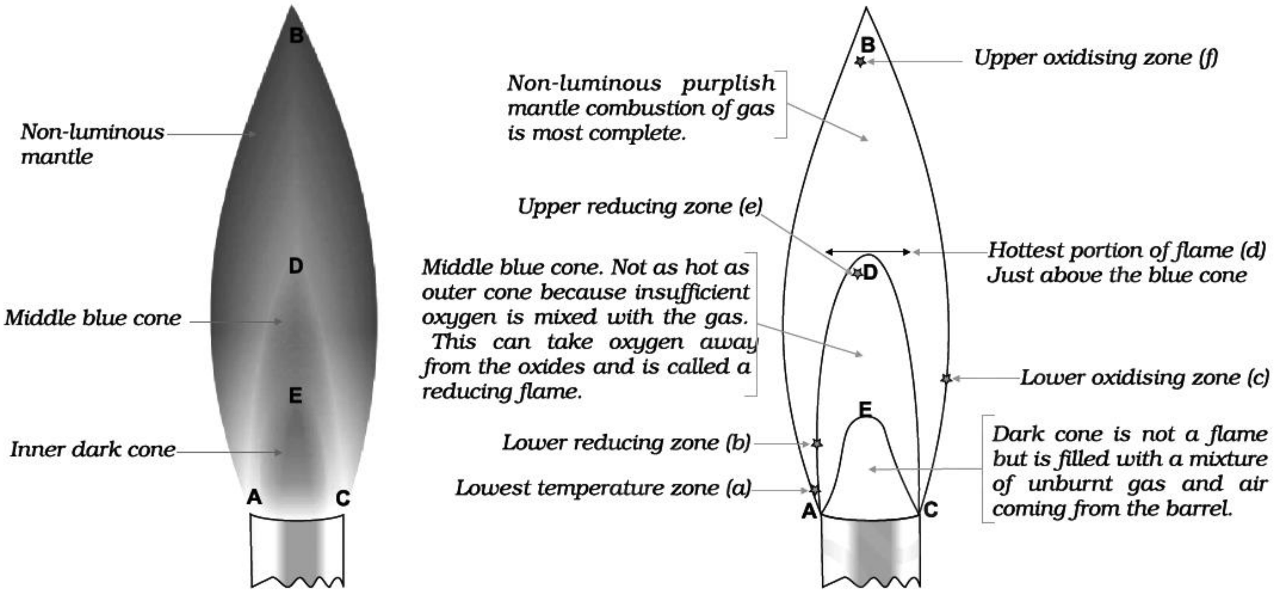


Fig. 1.13 : *Zones of flame of Bunsen burner*

(B) PRINCIPAL PARTS OF BUNSEN FLAME

1. The Inner Dark Cone, A E C

This is innermost dark cone, which is just above the burner tube. It consists of unburnt gases. This zone is the coldest zone of the flame and no combustion takes place here.

2. The Middle Blue Cone, A D C E A

This is middle part of the flame. This becomes luminous when the air vent is slightly closed. Luminosity of this part is due to the presence of unburnt carbon particles produced by decomposition of some gas. These particles get heated up to incandescence and glow but do not burn. Since the combustion is not complete in this part, the temperature is not very high.

3. The Outer Non-luminous Mantle, A B C D A

This is purplish outer cone. It is the hottest part of the flame. It is in direct contact with the atmosphere and combustion is quite complete in this zone.

Bunsen identified six different regions in these three principal parts of the flame:

(i) The upper oxidising zone (f)

Its location is in the non-luminous tip of the flame which is in the air. In comparison to inner portions of the flame large excess of oxygen is present here. The temperature is not as high as in region (c) described below. It may be used for all oxidation processes in which highest temperature of the flame is not required.

(ii) Upper reducing zone (e)

This zone is at the tip of the inner blue cone and is rich in incandescent carbon. It is especially useful for reducing oxide incrustations to the metals.

(iii) Hottest portion of flame (d)

It is the fusion zone. It lies at about one-third of the height of the flame and is approximately equidistant from inside and outside of the mantle i.e. the outermost cone of the flame. Fusibility of the substance can be tested in this region. It can also be employed for testing relative volatility of substances or a mixture of substances.

(iv) ***Lower oxidising zone (c)***

It is located on the outer border of the mantle near the lower part of the flame and may be used for the oxidation of substances dissolved in beads of borax or sodium carbonate etc.

(v) ***Lower reducing zone (b)***

It is situated in the inner edge of the outer mantle near to the blue cone and here reducing gases mix with the oxygen of the air. It is a less powerful reducing zone than (e) and may be employed for the reduction of fused borax and similar beads.

(vi) ***Lowest temperature zone (a)***

Zone (a) of the flame has lowest temperature. It is used for testing volatile substances to determine whether they impart colour to the flame.

(C) STRIKING BACK OF THE BUNSEN BURNER

Striking back is the phenomenon in which flame travels down the burner tube and begins to burn at the nozzle near the base. This happens when vents are fully open.The flow of much air and less gas makes the flame become irregular and it strikes back.

The tube becomes very hot and it may produce burns on touching. This may melt attached rubber tube also. If it happens, put off the burner and cool it under the tap and light it again by keeping the air vent partially opened.

SPIRIT LAMP

If Bunsen burner is not available in the laboratory then spirit lamp can be used for heating. It is a devise in which one end of a wick of cotton thread is dipped in a spirit container and the other end of the wick protrudes out of the nozzle at upper end of the container. Spirit rises upto the upper end of the wick due to the capillary action and can be burnt. The flame is non luminous hence can be used for all heating purposes in the laboratory. To put off the lamp, burning wick is covered with the cover. **Never try to put off the lighted burner by blowing at the flame.**

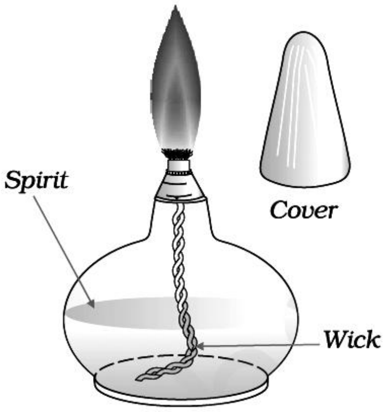


Fig. 1.14 : *The spirit lamp*

KEROSENE HEATING LAMP

A kerosene lamp has been developed by National Council of Educational Research and Training (NCERT), which is a versatile and cheaper substitute of spirit lamp. It may be used in laboratories as a source of heat whereever spirit and gas burner are not available. Parts of kerosene lamp are shown in Fig.

Working of the Kerosene Lamp

More than half of the container is filled with kerosene. Outer sleeve is removed for lighting the wicks. As the outer sleeve is placed back in position, the flames of four wicks combine to form a big soot-free blue flame.

The lighted heating lamp can be put off only by covering the top of the outer sleeve with a metal or asbestos sheet.

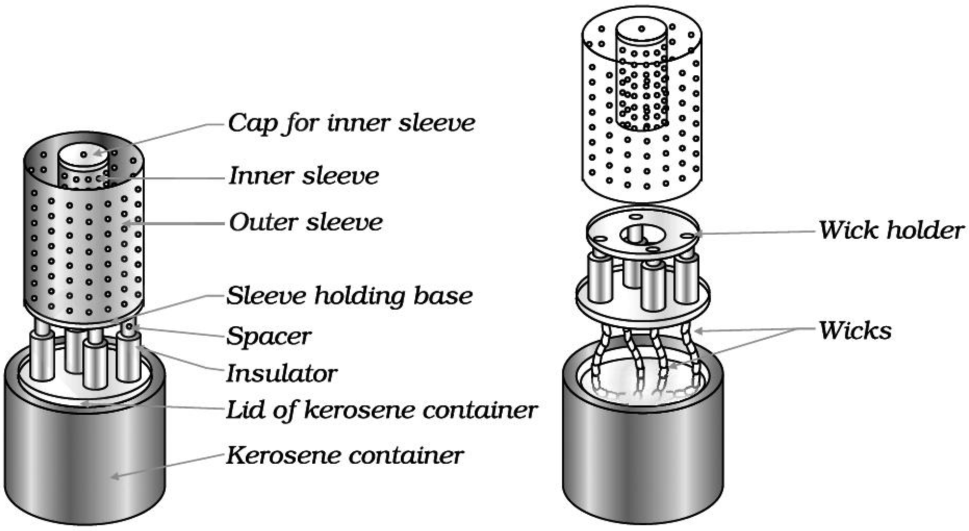


Fig. 1.15 : *Parts of Kerosene Heating Lamp*

SYSTEMATIC ANALYSIS OF CATIONS

The tests for cations may be carried out according to the following scheme.

Step - I : Preliminary Examination of the Salt for Identification of Cation

1. Colour Test

Observe the colour of the salt carefully, which may provide useful information about the cations. Table 6 gives the characteristic colours of the salts of some cations.

Table 6 Characteristic colours of the some metal ions

Ion	Confirmatory Test
Light green	Fe^{2+}
Yellowis Brown	Fe^{3+}
Blue	Cu^{2+}
Bright green	Ni^{2+}
Blue, Red Violet, Pink	Co^{2+}
Light pink	Mn^{2+}

2. Dry Heating Test

(i) Take about 0.1 g of the dry salt in a clean and dry test tube.

(ii) Heat the above test tube for about one minute and observe the colour of the residue when it is hot and also when it becomes cold. Observation of changes gives indications about the presence of cations, which may not be taken as conclusive evidence (see Table 7).

Table 7 : Inferences from the colour of the salt in cold and on heating

Colour when cold	Colour when hot	Inference
Blue	White	Cu^{2+}
Green	Dirty white or yellow	Fe^{2+}
White	Yellow	Zn^{2+}
Pink	Blue	Co^{2+}

3. Flame Test

The chlorides of several metals impart characteristic colour to the flame because they are volatile in non-luminous flame. This test is performed with the help of a platinum wire as follows :

(i) Make a tiny loop at one end of a platinum wire.

(ii) To clean the loop dip it into concentrated hydrochloric acid and hold it in a non-luminous flame (Fig. 1.3).

(iii) Repeat step (ii) until the wire imparts no colour to the flame.

(iv) Put 2-3 drops of concentrated hydrochloric acid on a clean watch glass and make a paste of a small quantity of the salt in it.

(v) Dip the clean loop of the platinum wire in this paste and introduce the loop in the non-luminous (oxidising) flame (Fig. 1.3).

(vi) Observe the colour of the flame first with the naked eye and then through a blue glass and identify the metal ion with the help of Table 8.

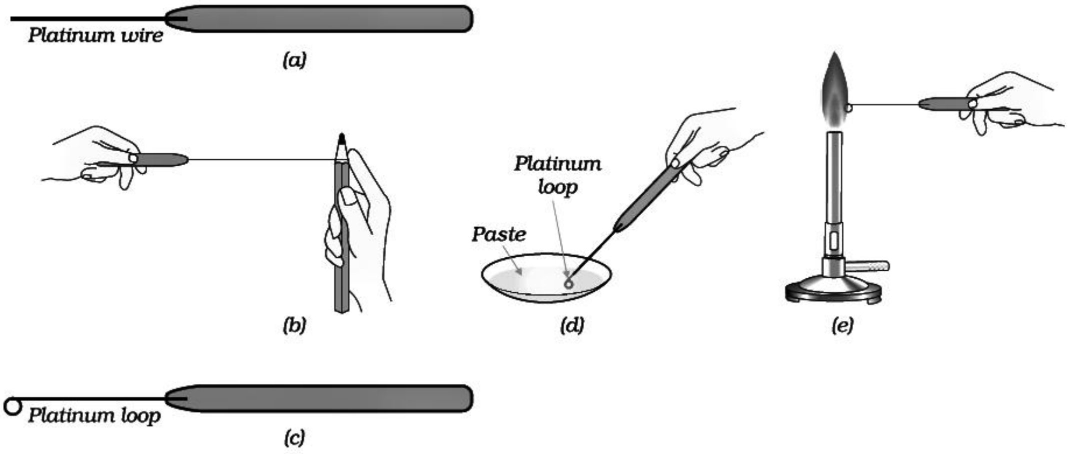


Fig. 1.3 : Performing flame test

Table 8 : Inference from the flame test

Colour fo the flame observed by naked eye	Colour of the flame observed through blue glass	Inference
Green flame with blue centre	Same colour as observed without glass	Cu^{2+}
Crimson red	Purple	Sr^{2+}
Apple green	Bluish green	Ba^{2+}
Brick red	Green	Ca^{2+}

4. Borax Bead Test

This test is employed only for coloured salts because borax reacts with metal salts to form metal borates or metals, which have characteristic colours.

(i) To perform this test make a loop at the end of the platinum wire and heat it in a flame till it is red hot.

(ii) Dip the hot loop into borax powder and heat it again until borax forms a colourless transparent bead on the loop. Before dipping the borax bead in the test salt or mixture, confirm that the bead is transparent and colourless. If it is coloured this means that, the platinum wire is not clean. Then make a fresh bead after cleaning the wire.

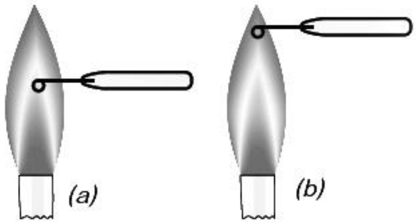


Fig. 1.4 : Borax bead test (a) Heating in reducing flame (b) Heating in oxidising flame

(iii) Dip the bead in a small quantity of the dry salt and again hold it in the flame.

(iv) Observe the colour imparted to the bead in the non - luminous flame as well as in the luminous flame while it is hot and when it is cold (Fig. 1.4).

(v) To remove the bead from the platinum wire, heat it to redness and tap the platinum wire with your finger. (Fig.1.5).

On heating, borax loses its water of crystallisation and decomposes to give sodium metaborate and boric anhydride.

$$\underset{\text{Borax}}{Na_2B_4O_7 \cdot 10H_2O} \longrightarrow Na_2B_4O_7 + 10H_2O$$

$$Na_2B_4O_7 \longrightarrow \underset{\text{Sodium metaborate}}{2NaBO_2} + \underset{\text{Boric anhydride}}{B_2O_3}$$

On treatment with metal salt, boric anhydride forms metaborate of the metal which gives different colours in oxidising and reducing flame. For example, in the case of copper sulphate, following reactions occur.

$$CuSO_4 + B_2O_3 \xrightarrow{\text{Non-luminous flame}} \underset{\substack{\text{Cupric metaborate} \\ \text{Blue-green}}}{Cu(BO_2)_2} + SO_3$$

Two reactions may take place in the reducing flame:

(i) The blue $Cu(BO_2)_2$ is reduced to colourless cuprous metaborate as follows:

$$2Cu(BO_2)_2 + 2NaBO_2 + C \xrightarrow{\text{Luminous flame}} 2CuBO_2 + Na_2B_4O_7 + CO$$

or (ii) Cupric metaborate may be reduced to metallic copper and the bead appears red and opaque.

$$2Cu(BO_2)_2 + 4NaBO_2 + 2C \xrightarrow{\text{Luminous flame}} 2Cu + 2Na_2B_4O_7 + 2CO$$

The preliminary identification of metal ion can be made from Table 9.

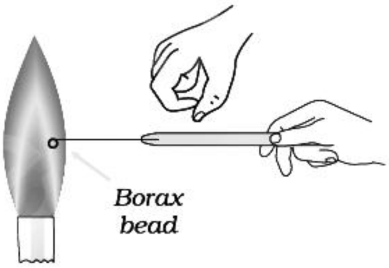


Fig. 1.5 : Removing borax bead

Table 9 : Inference from the borax bead test

Heating in oxidising (non-luminous) flame		Heating in reducing (luminous) flame		Inference
Colour of the salt bead		**Colour of the salt bead**		
In cold	**In hot**	**In cold**	**In hot**	
Blue	Gren	Red opaque	Colourless	Cu^{2+}
Reddish brown	Violet	Grey	Grey	Ni^{2+}
Light violet	Light violet	Colourless	Colourless	Mn^{2+}
Yellow	Yellowish brown	Green	Green	Fe^{3+}

5. Charcoal Cavity Test

Metallic carbonate when heated in a charcoal cavity decomposes to give corresponding oxide. The oxide appears as a coloured residue in the cavity. Sometimes oxide may be reduced to metal by the carbon of the charcoal cavity.

The test may be performed as follows:

(i) Make a small cavity in a charcoal block with the help of a charcoal borer [Fig.1.6 (a)].

(ii) Fill the cavity with about 0.2 g of the salt and about 0.5 g of anhydrous sodium carbonate.

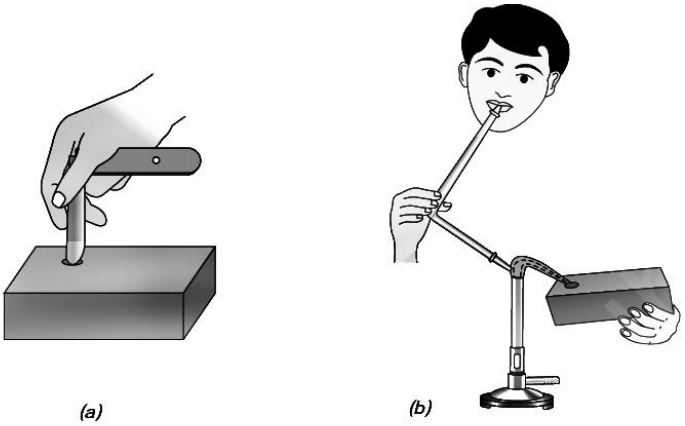


Fig. 1.6 : (a) Making charcoal cavity (b) Heating salt in the cavity

(iii) Moisten the salt in the cavity with one or two drops of water, otherwise salt/mixture will blow away.

(iv) Use a blowpipe to heat the salt in a luminous (reducing) flame and observe the colour of oxide/ metallic bead formed in the cavity both when hot and cold [Fig. (1.6 b)]. Obtain oxidising and reducing flame as shown in Fig. 1.7 a and b.

(v) Always bore a fresh cavity for testing the new salt.

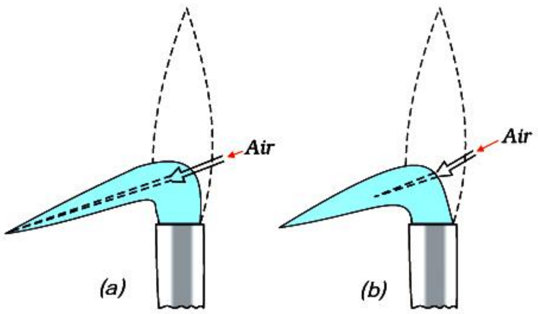


Fig. 1.7 : Obtaining oxidising and reducing flame (a) Oxidising flame; and (b) Reducing flame

When test is performed with $CuSO_4$, the following change occurs.

$$CuSO_4 + Na_2CO_3 \xrightarrow{Heat} CuCO_3 + Na_2SO_4$$

$$CuCO_3 \xrightarrow{Heat} CuO + CO_2$$

$$CuO + C \xrightarrow{Heat} \underset{\text{Red colour}}{Cu} + CO$$

In case of $ZnSO_4$:

$$ZnSO_4 + Na_2CO_3 \xrightarrow{Heat} ZnCO_3 + Na_2SO_4$$

$$ZnCO_3 \xrightarrow{Heat} \underset{\substack{\text{Yellow when hot,}\\ \text{White when cold}}}{ZnO} + CO_2$$

The metal ion can be inferred from Table 10.

Table 10 : Inference from the charcoal cavity test

Observations	Inference
Yellow residue when hot and grey metal when cold	Pb^{2+}
White residue with the odour of garlic	As^{3+}
Brown residue	Cd^{2+}
Yellow residue when hot and white when cold	Zn^{2+}

6. Cobalt Nitrate Test

If the residue in the charcoal cavity is white, cobalt nitrate test is performed.

(i) Treat the residue with two or three drops of cobalt nitrate solution.

(ii) Heat it strongly in non-luminous flame with the help of a blow pipe and observe the colour of the residue.

On heating, cobalt nitrate decomposes into cobalt (II) oxide, which gives a characteristic colour with metal oxide present in the cavity.

Thus, with ZnO, Al_2O_3 and MgO, the following reactions occur.

$$2Co(NO_3)_2 \xrightarrow{Heat} 2CoO + 4NO_2 + O_2$$

$$CoO + ZnO \longrightarrow \underset{\text{Green}}{CoO.ZnO}$$

$$CoO + MgO \longrightarrow \underset{\text{Pink}}{CoO.\ MgO}$$

$$CoO + Al_2O_3 \longrightarrow \underset{\text{Blue}}{CoO.\ Al_2O_3}$$

Step-II : Wet Tests for Identification of Cations

The cations indicated by the preliminary tests given above are confirmed by systematic analysis given below.

The first essential step is to prepare a clear and transparent solution of the salt. This is called original solution. It is prepared as follows:

Preparation of Original Solution (O.S.)

To prepare the original solution, following steps are followed one after the other in a systematic order. In case the salt does not dissolve in a particular solvent even on heating, try the next solvent.

The following solvents are tried:

1. Take a little amount of the salt in a clean boiling tube and add a few mL of distilled water and shake it. If the salt does not dissolved, heat the content of the boiling tube till the salt completely dissolves.
2. If the salt is insoluble in water as detailed above, take fresh salt in a clean boiling tube and add a few mL of dil.HCl to it. If the salt is insoluble in cold, heat the boiling tube till the salt is completely dissolved.
3. If the salt does not dissolve either in water or in dilute HCl even on heating, try to dissolve it in a few mL of conc. HCl by heating.
4. If salt does not dissolve in conc. HCl, then dissolve it in dilute nitric acid.
5. If salt does not dissolve even in nitric acid then a mixture of conc. HCl and conc. HNO_3 in the ratio 3 : 1 is tried. This mixture is called aqua regia. A salt not soluble in aqua regia is considered to be an insoluble salt.

Group Analysis

(I) Analysis of Zero group cation (NH_4^+ ion)

(a) Take 0.1 g of salt in a test tube and add 1-2 mL of NaOH solution to it and heat. If there is a smell of ammonia, this indicates the presence of ammonium ions. Bring a glass rod dipped in hydrochloric acid near the mouth of the test tube. White fumes are observed.

(b) Pass the gas through Nessler's reagent. Brown precipitate is obtained.

Chemistry of Confirmatory Tests for NH_4^+ ion

(a) Ammonia gas evolved by the action of sodium hydroxide on ammonium salts reacts with hydrochloric acid to give ammonium chloride, which is visible as dense white fume.

$$(NH_4)_2SO_4 + 2NaOH \longrightarrow Na_2SO_4 + 2NH_3 + 2H_2O$$

$$NH_3 + HCl \longrightarrow NH_4Cl$$

On passing the gas through Nessler's reagent, a brown colouration or a precipitate of basic mercury(II) amido-iodine is formed.

$$2K_2[HgI_4] + NH_3 + 3KOH \longrightarrow \underset{\substack{\text{Basic mercury (II)}\\ \text{amido-iodine}\\ \text{(Brown precipitate)}}}{HgO.Hg(NH_2)I} + 7KI + 2H_2O$$

For the analysis of cations belonging to groups I-VI, the cations are precipitated from the original solution by using the group reagents (see Table 1.11) according to the scheme shown in the flow chart given below:

The separation of all the six groups is represented as below :

Flow Chart*
Original Solution

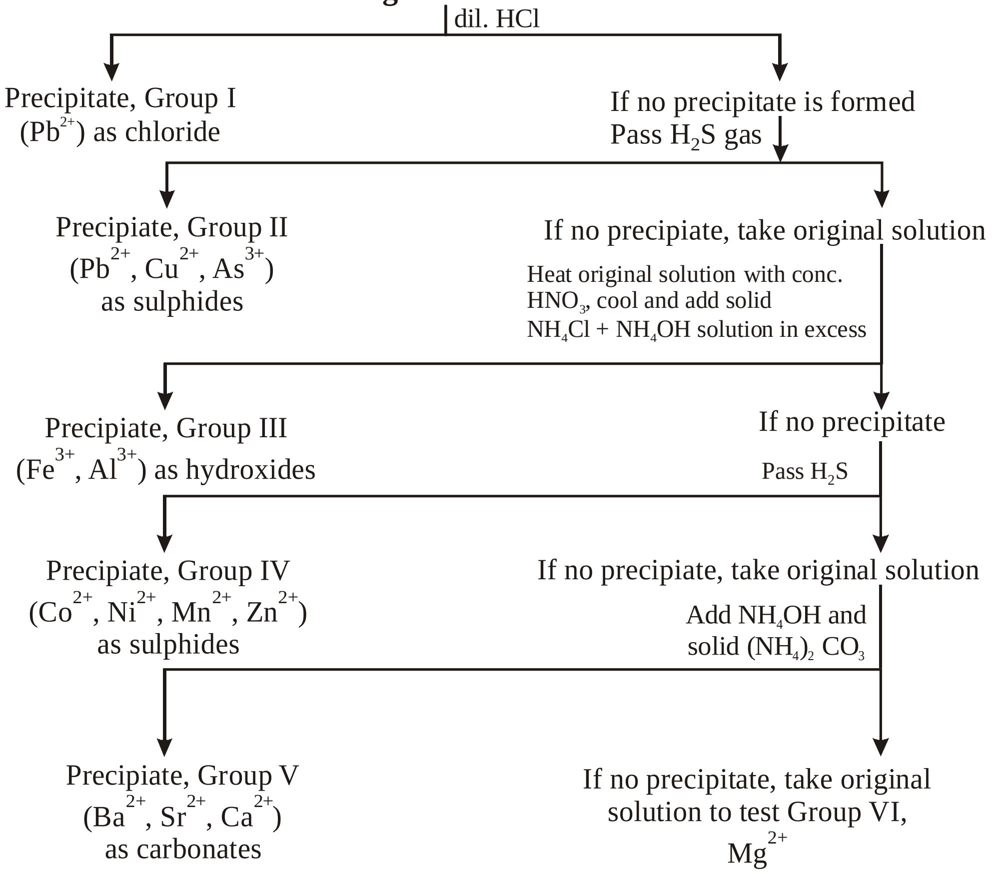


Table 11 : Group reagents for precipitating ions

Group	Cations*	Group Reagent
Group zero	NH_4^+	None
Group - I	Pb^{2+}	Dilute HCl
Group - II	Pb^{2+}, Cu^{2+}, As^{3+}	H_2S gas in presence of dil. HCl
Group - III	Al^{3+}, Fe^{3+}	NH_4OH in presence of NH_4Cl
Group - IV	Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+}	H_2S in presence of NH_4OH
Group - V	Ba^{2+}, Sr^{2+}, Ca^{2+}	$(NH_4)_2CO_3$ in presence of NH_4OH
Group - VI	Mg^{2+}	None

(II) Analysis of Group-I cations

Take a small amount of original solution (if prepared in hot conc. HCl) in a test tube and add cold water to it and cool the test tube under tap water. If a white precipitate appears, this indicates the presence of Pb^{2+} ions in group –I. On the other hand, if the original solution is prepared in water and on addition of dil. HCl, a white precipitate appears, this may also be Pb^{2+}. Confirmatory tests are described below in Table 12.

Table 12 : Confirmatory tests for Group-I cation (Pb^{2+})

Experiment	Observation
Dissolve the precipitate in hot water and divide the hot solution into three parts,	
1. Add potassium idoide solution to the first part.	A yellow precipitate is obtained.
2. To the second part add potassium chromate solution.	A yellow precipitate is obtained which is soluble, in NaOH and insoluble in ammonium acetate solution.
3. To the thrid part of the hot solution add few drops of alcohol and dilute sulphuric acid.	A white precipitate is obtained which is soluble in ammonium acetate solution.

Chemistry of the Confirmatory Tests of Pb^{2+} ions

Lead is precipitated as lead chloride in the first group. The precipitate is soluble in hot water.

1. On adding potassium iodide (KI) solution, a yellow precipitate of lead iodide is obtained which confirms the presence of Pb^{2+} ions.

$$\underset{\text{(Hot solution)}}{PbCl_2} + 2KI \longrightarrow \underset{\text{Yellow precipitate}}{PbI_2} + 2KCl$$

This yellow precipitate (PbI_2) is soluble in boiling water and reappears on cooling as shining crystals.

2. On addition of potassium chromate (K_2CrO_4) solution a yellow precipitate of lead chromate is obtained. This confirms the presence of Pb^{2+} ions.

$$\underset{\text{(Hot solution)}}{PbCl_2} + K_2CrO_4 \longrightarrow \underset{\substack{\text{Lead chromate}\\\text{(Yellow precipitate)}}}{PbCrO_4} + 2KCl$$

The yellow precipitate ($PbCrO_4$) is soluble in hot NaOH solution.

$$PbCrO_4 + 4NaOH \longrightarrow \underset{\substack{\text{Sodium tetra}\\\text{hydroxoplumbate (II)}}}{Na_2[Pb(OH)_4]} + Na_2CrO_4$$

3. A white precipitate of lead sulphate ($PbSO_4$) is formed on addition of alcohol followed by dil. H_2SO_4.

$$PbCl_2 + H_2SO_4 \longrightarrow \underset{\substack{\text{Lead sulphate}\\\text{(White precipitate)}}}{PbSO_4} + 2HCl$$

Lead sulphate is soluble in ammonium acetate solution due to the formation of tetraacetatoplumbate(II) ions. This reaction may be promoted by addition of few drops of acetic acid.

$$PbSO_4 + 4CH_3COONH_4 \longrightarrow \underset{\substack{\text{Ammonium}\\\text{tetraacetatoplumbate(II)}}}{(NH_4)_2[Pb(CH_3COO)_4]} + (NH_4)_2SO_4$$

(III) Analysis of Group–II cations

If group-I is absent, add excess of water to the same test tube. Warm the solution and pass H_2S gas for 1-2 minutes (Fig. 1.6). Shake the test tube. If a precipitate appears, this indicates the presence of group-II cations. Pass more H_2S gas through the solution to ensure complete precipitation and separate the precipitate. If the colour of the precipitate is black, itindicates the presence of Cu^{2+} or Pb^{2+} ions.

If it is yellow in colour, then presence of As^{3+} ions is indicated.

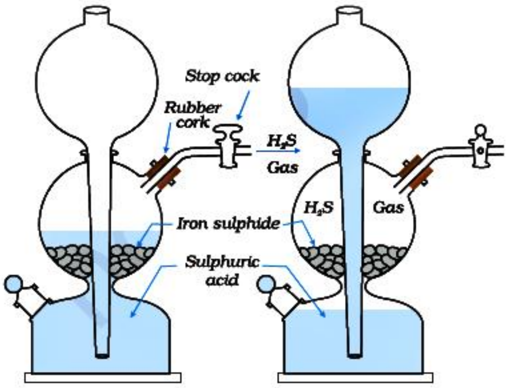


Fig. 1.8 : Kipp's apparatus for preparation of H_2S gas

Take the precipitate of group-II in a test tube and add excess of yellow ammonium sulphide solution to it. Shake the test tube. If the precipitate is insoluble, group II-A (copper group) is present. If the precipitate is soluble, this indicates the presence of group-II B (arsenic group).

Confirmatory tests for the groups II A and II B are given in Table 13.

Table 13 : Confirmatory tests for the groups II A and II B cations

Black precipitate of Group II A ions Pb^{2+}, Cu^{2+} (insoluble in yellow ammonium sulphide) is formed.		If a yellow precipitate soluble in yellow ammonium sulphide is formed then As^{3+} ion is present.
Boil the precipitate of Group II A with dilute nitric acid and add a few drops of alcohol and dil. H_2SO_4.		Acidify this solution with dilute HCl. A yellow precipitate is formed. Heat the precipitate with concentrated nitric acid and add ammonium molybdate solution. A canary yellow precipitate is formed.
White precipitate confirms the presence of Pb^{2+} ions. Dissolve the precipitate in ammonium acetate solution. Acidify with acetic acid and divide the solution into two parts. (i) To the first part add potassium chromate solution, a yellow precipitate is formed. (ii) To the second part, add potassium iodide solution, a yellow precipitate is formed.	If no precipitate is formed, add excess of ammonium hydroxide solution. A blue solution is obtained, acidify it with acetic acid and add potassium ferrocyanide solution. A chocolate brown precipitate is formed.	

Group-II A (Copper Group)

Chemistry of confirmatory tests of Group-II A cations

1. Test for Lead ion (Pb^{2+})

Lead sulphide precipitate dissolves in dilute HNO_3. On adding dil. H_2SO_4 and a few drops of alcohol to this solution a white precipitate of lead sulphate appears. This indicates the presence of lead ions.

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

$$Pb(NO_3)_2 + H_2SO_4 \longrightarrow PbSO_4 + 2HNO_3$$

The white precipitate dissolves in ammonium acetate solution on boiling. When this solution is acidified with acetic acid and potassium chromate solution is added, a yellow precipitate of $PbCrO_4$ is formed. On adding potassium iodide solution, a yellow precipitate of lead iodide is formed.

$$PbSO_4 + 4CH_3COONH_4 \longrightarrow \underset{\substack{\text{Ammonium}\\\text{tetraacetatoplumbate(II)}}}{(NH_4)_2[Pb(CH_3COO)_4]} + (NH_4)_2SO_4$$

$$Pb^{2+} + CrO_4^{2-} \longrightarrow \underset{\substack{\text{Lead chromate}\\\text{(Yellow precipitate)}}}{PbCrO_4} ; \quad \underset{\substack{\text{Lead iodide}\\\text{(Yellow precipitate)}}}{Pb^{2+} + 2I^- \rightarrow PbI_2}$$

2. Test for Copper ion (Cu^{2+})

(a) Copper sulphide dissolves in nitric acid due to the formation of copper nitrate.

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

On heating the reaction mixture for long time, sulphur is oxidised to sulphate and copper sulphate is formed and the solution turns blue. A small amount of NH_4OH precipitates basic copper sulphate which is soluble in excess of ammonium hydroxide due to the formation of tetraamminecopper (II) complex.

$$S + 2HNO_3 \longrightarrow H_2SO_4 + 2NO$$

$$2Cu^{2+} + SO_4^{2-} + 2NH_3 + 2H_2O \longrightarrow Cu(OH)_2.CuSO_4 + 2NH_4^+$$

$$Cu(OH)_2.CuSO_4 + 8NH_3 \longrightarrow \underset{\substack{\text{Tetraamminecopper (II)}\\\text{sulphate (Deep blue)}}}{2[Cu(NH_3)_4]SO_4} + 2OH^- + SO_4^{2-}$$

(b) The blue solution on acidification with acetic acid and then adding potassium ferrocyanide $K_4[Fe(CN)_6]$ solution gives a chocolate colouration due to the formation of copper ferrocyanide

i.e. $Cu_2[Fe(CN)_6]$.

$$[Cu(NH_3)_4]SO_4 + 4CH_3COOH \longrightarrow CuSO_4 + 4CH_3COONH_4$$

$$2CuSO_4 + \underset{\substack{\text{Potassium}\\\text{hexacyanoferrate (II)}}}{K_4[Fe(CN)_6]} \longrightarrow \underset{\substack{\text{Copper}\\\text{hexacyanoferrate (II)}\\\text{(Chocolate brown precipitate)}}}{Cu_2[Fe(CN)_6]} + 2K_2SO_4$$

Group-II B (Arsenic Group)

If group- II precipitate dissolves in yellow ammonium sulphide and the colour of the solution is yellow, this indicates the presence of As^{3+} ions. Ammonium thioarsenide formed on dissolution of As_2S_3, decomposes with dil. HCl, and a yellow precipitate of arsenic (V) sulphide is formed which dissolves in concentrated nitric acid on heating due to the formation of arsenic acid. On adding ammonium molybdate solution to the reaction mixture and heating, a canary yellow precipitate is formed. This confirms the presence of As^{3+} ions.

$$As_2S_3 + \underset{\substack{\text{Yellow ammonium}\\ \text{sulphide}}}{3(NH_4)_2S} \longrightarrow 2(NH_4)_3AsS_4 + S$$

$$2(NH_4)_3AsS_4 + 6HCl \rightarrow As_2S_5 + 3H_2S + 6NH_4Cl$$

$$3As_2S_5 + 10HNO_3 + 4H_2O \rightarrow \underset{\text{Arsenic acid}}{6H_3AsO_4} + 10NO + 15S$$

$$\underset{\text{Arsenic acid}}{H_3AsO_4} + \underset{\substack{\text{Ammonium}\\ \text{molybdate}}}{12(NH_4)_2MoO_4} + 21HNO_3 \rightarrow \underset{\substack{\text{Ammonium}\\ \text{arsenomolybdate}\\ \text{(yellow precipitate)}}}{(NH_4)_3[As\ (Mo_3O_{10})_4]} + 21NH_4NO_3 + 12H_2O$$

(IV) Analysis of Group–III cations

If group-II is absent, take original solution and add 2-3 drops of conc. HNO_3 to oxidise Fe^{2+} ions to Fe^{3+} ions. Heat the solution for a few minutes. After cooling add a small amount of solid ammonium chloride (NH_4Cl) and an excess of ammonium hydroxide (NH_4OH) solution till it smells of ammonia. Shake the test tube. If a brown or white precipitate is formed, this indicates the presence of group-III cations. Confirmatory tests of group-III cations are summarised in Table 14.

Observe the colour and the nature of the precipitate. A gelatinous white precipitate indicates the presence of aluminium ion ($A1^{3+}$). If the precipitate is brown in colour, this indicates the presence of ferric ions (Fe^{3+}).

Table 14 : Confirmatory test for Group-III cations

	Brown precipitate **Fe^{3+}**		**White precipitate** **Al^{3+}**
	Dissolve the precipitate in dilute HCl and divide the solution into two parts.		Dissolve the white precipitate in dilute HCl and divide into two parts. .
(a)	To the first part add potassium ferrocyanide solution [Potasium hexacyanoferrate (II)]. A blue precipitate/colouration appears.	(a)	To the first part add sodium hydroxide solution and warm. A white gelatinous precipitate soluble in excess of sodium hydroxide solution is obtained
(b)	To the second part add potassium thiocyanate solution. A blood red colouration appears.	(b)	To the second part first add blue litmus solution and then ammonium hydroxide solution drop by drop along the sides of the test tube. A blue floating mass in the colourless solution is obtained.

Chemistry of confirmatory tests of Group - III cations

When original solution is heated with concentrated nitric acid, ferrous ion are oxidised to ferric ions.

$$2FeCl_2 + 2HCl + [O] \longrightarrow 2FeCl_3 + H_2O$$

Their group cations are precipitated as their hydroxides, which dissolve in dilute hydrochloric acid due to the formation of corresponding chlorides.

1. Test for Aluminium ions (Al^{3+})

(a) When the solution containing aluminium chloride is treated with sodium hydroxide a white gelationus precipitate of aluminium hydroxide is formed which is soluble in excess of sodium hydroxide solution due to the formation of sodium meta aluminate.

$$AlCl_3 + 3NaOH \longrightarrow Al(OH)_3 + 3NaCl$$

$$\underset{\text{White gelatinous precipitate}}{Al(OH)_3} + NaOH \longrightarrow \underset{\text{Sodium meta aluminate}}{NaAlO_2} + 2H_2O$$

(b) In the second test when blue litmus is added to the solution, a red colouration is obtained due to the acidic nature of the solution. On addition of NH_4OH solution drop by drop, the solution becomes alkaline and aluminium hydroxide is precipitated. Aluminium hydroxide adsorbs blue colour from the solution and forms insoluble adsorption complex named 'lake'. Thus a blue mass floating in the colourless solution is obtained. The test is therefore called **lake test.**

2. Test for ferric ions (Fe^{3+})

Reddish brown precipitate of ferric hydroxide dissolves in hydrochloric acid and ferric chloride is formed.

$$Fe(OH)_3 + 3HCl \longrightarrow FeCl_3 + 3H_2O$$

(a) When the solution containing ferric chloride is treated with potassium ferrocyanide solution a blue precipitate/colouration is obtained. The colour of the precipitate is Prussian blue. It is ferric ferrocyanide. The reaction takes place as follows:

$$4FeCl_3 + \underset{\text{Potassium ferrocyanide}}{3K_4[Fe(CN)_6]} \longrightarrow \underset{\text{Prussian blue precipitate}}{Fe_4[Fe(CN)_6]_3} + 12KCl$$

If potassium hexacyanoferrate (II) (i.e. potassium ferrocyanide) is added in excess then a product of composition $KFe[Fe(CN)_6]$ is formed. This tends to form a colloidal solution ('soluble Prussian blue') and cannot be filtered.

$$FeCl_3 + K_4[Fe(CN)_6] \longrightarrow \underset{\text{(Soluble prussian blue)}}{KFe[Fe(CN)_6]} + 3KCl$$

(b) To the second part of the solution, add potassium thiocyanate (potassium sulphocyanide) solution. The appearance of a blood red colouration confirms the presence of Fe^{3+} ions.

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

(V) Analysis of group-IV cations

If group-III is absent, pass H_2S gas in the solution of group-III for a few minutes. If a precipitate appears (white, black or flesh coloured), this indicates the presence of group-IV cations. Table 15 gives a summary of confirmatory tests of group-IV cations.

Table 15 : Confirmatory test for Group - IV cations

White precipitate (Zn^{2+})	**Flesh coloured precipitate (Mn^{2+})**	**Black precipitate (Ni^{2+}, Co^{2+})**
Dissolve the precipitate in dilute HCl by boiling. Divide the solution into two parts. (a) To the first part add sodium hydroxide solution. A white precipitate soluble in excess of sodium hydroxide solution confirms the presence of Zn^{2+} ions. (b) Neutralise the second part with a mmonium hydroxide solution and add potassium ferrocyanide solution. A bluish white precipitate appears	Dissolve the precipitate in dilute HCl by boiling, then add sodium hydroxide solution in excess. A white precipitate is formed which turns brown on keeping.	Dissolve the precipitate in aqua regia. Heat the solution to dryness and cool. Dissolve the residue in water and divide the solution into two parts. (a) To the first part of the solution add ammonium hydroxide solution till it becomes alkaline. Add a few drops of dimethyl glyoxime and shake the test tube. Formation of a bright red precipitate confirms the presence of Ni^{2+} ions. (b) Neutralise the second part with ammonium hydroxide solution. Acidify it with dilute acetic acid and add solid potassium nitrite. A yellow precipitate confirms the presence of Co^{2+} ions.

Chemistry of confirmatory tests of Group–IV cations

Fourth group cations are precipitated as their sulphides. Observe the colour of the precipitate. A white colour of the precipitate indicates the presence of zinc ions, a flesh colour indicates the presence of manganese ions and a black colour indicates the presence of Ni^{2+} or Co^{2+} ions.

1. Test for Zinc ion (Zn^{2+})

Zinc sulphide dissolves in hydrochloric acid to form zinc chloride.

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

(a) On addition of sodium hydroxide solution it gives a white precipitate of zinc hydroxide, which is soluble in excess of NaOH solution on heating. This confirms the presence of Zn^{2+} ions.

$$ZnCl_2 + 2NaOH \longrightarrow Zn(OH)_2 + 2NaCl$$

$$Zn(OH)_2 + 2NaOH \longrightarrow \underset{\text{Sodium zincate}}{Na_2ZnO_2} + 2H_2O$$

(b) When potassium ferrocyanide $K_4[Fe(CN)_6]$ solution is added to the solution after neutralisation by NH_4OH solution, a white or a bluish white precipitate of zinc ferrocyanide appears.

$$2ZnCl_2 + K_4[Fe(CN)_6] \longrightarrow \underset{\text{Zinc ferrocyanide}}{Zn_2[Fe(CN)_6]} + 4KCl$$

2. Test for Manganese ion (Mn^{2+})

Manganese sulphide precipitate dissolves in dil. HCl on boiling. On addition of NaOH solution in excess, a white precipitate of manganese hydroxide is formed which turns brown due to atmospheric oxidation into hydrated manganese dioxide.

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

$$MnCl_2 + 2NaOH \longrightarrow \underset{\text{(White precipitate)}}{Mn(OH)_2} + 2NaCl$$

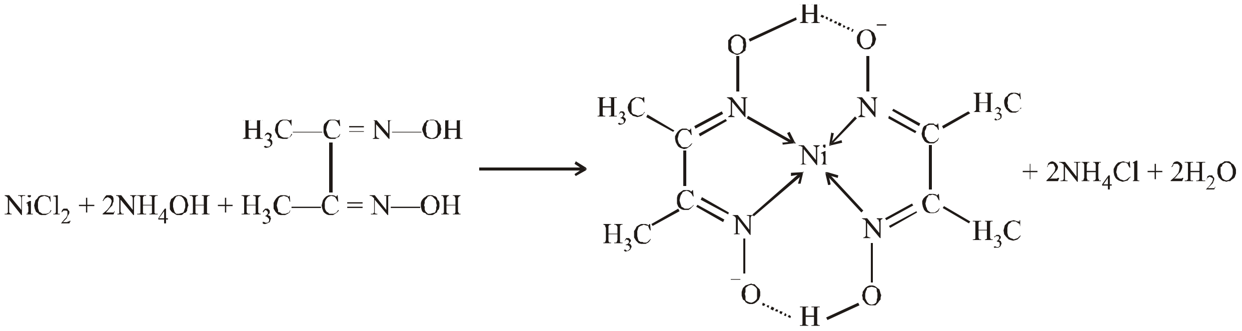
$$Mn(OH)_2 + [O] \longrightarrow \underset{\substack{\text{Hydrated manganese dioxide} \\ \text{(Brown colour)}}}{MnO(OH)_2}$$

3. Test for Nickel ion (Ni^{2+})

The black precipitate of nickel sulphide dissolves in aqua regia and the reaction takes place as follows:

$$3NiS + 2HNO_3 + 6HCI \longrightarrow 3NiCl_2 + 2NO + 3S + 4H_2O$$

After treatment with aqua regia nickel-chloride is obtained which is soluble in water. When dimethyl glyoxime is added to the aqueous solution of nickel chloride, made alkaline, by adding NH_4OH solution, a brilliant red precipitate is obtained.



Complex of red colour
(Stable form of complex)

4. Test for Cobalt ion (Co^{2+})

Cobalt sulphide dissolves in aqua regia in the same manner as nickel sulphide. When the aqueous solution of the residue obtained after treatment with aqua regia is treated with a strong solution of potassium nitrite after neutralisation with ammonium hydroxide and the solution is acidified with dil. acetic acid, a yellow precipitate of the complex of cobalt named potassium hexanitritocobaltate (III) is formed.

$$CoS + HNO_3 + 3HCl \longrightarrow CoCl_2 + NOCl + S + 2H_2O$$

$$CoCl_2 + 7KNO_2 + 2CH_3COOH \longrightarrow \underset{\substack{\text{Potassium} \\ \text{hexanitritocobaltate(III)} \\ \text{(Yellow precipitate)}}}{K_3[Co(NO_2)_6]} + 2KCl + 2CH_3COOK + NO + H_2O$$

(VI) Analysis of Group–V cations

If group-IV is absent then take original solution and add a small amount of solid NH_4Cl and an excess of NH_4OH solution followed by solid ammonium carbonate $(NH_4)_2CO_3$. If a white precipitate appears, this indicates the presence of group–V cations.

Dissolve the white precipitate by boiling with dilute acetic acid and divide the solution into three parts one each for Ba^{2+}, Sr^{2+} and Ca^{2+} ions. Preserve a small amount of the precipitate for flame test. Summary of confirmatory tests is given in Table 16.

16 : Confirmatory test for Group - V cations

Dissolve the precipitate by boiling with dilute acetic acid and divide the solution into three parts one each for Ba^{2+}, Sr^{2+} and Ca^{2+} ions		
Ba^{2+} ions	**Sr^{2+} ions**	**Ca^{2+} ions**
(a) To the first part add potassium chromate solution. A yellow precipitate appears. (b) Perform the flame test with the preserved precipitate. A grassy green flame is obtained.	(a) If barium is absent, take second part of the solution and add ammonium sulphate solution. Heat and scratch the sides of the test tube with a glass rod and cool. A white precipitate is formed. (b) Perform the flame test with the preserved precipitate. A crimson-red flame confirms the presence of Sr^{2+} ions.	(a) If both barium and strontium are absent, take the third part of the solution. Add ammonium oxalate solution and shake well. A white precipitate of calcium oxalate is obtained. (b) Perform the flame test with the preserved precipitate. A brick red flame, which looks greenish-yellow through blue glass, confirms the presence of Ca^{2+} ions.

Chemistry of Confirmatory Tests of Group–V cations

The Group–V cations are precipitated as their carbonates which dissolve in acetic acid due to the formation of corresponding acetates.

1. Test for Barium ion (Ba^{2+})

(a) Potassium chromate (K_2CrO_4) solution gives a yellow precipitate of barium chromate when the solution of fifth group precipitate in acetic acid is treated with it.

$$BaCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ba + H_2O + CO_2$$

$$(CH_3COO)_2Ba + K_2CrO_4 \longrightarrow \underset{\substack{\text{Barium chromate}\\ \text{(yellow precipitate)}}}{BaCrO_4} + 2CH_3COOK$$

(b) **Flame test :** Take a platinum wire and dip it in conc. HCl. Heat it strongly until the wire does not impart any colour to the non-luminous flame. Now dip the wire in the paste of the (Group–V) precipitate in conc. HCl. Heat it in the flame. A grassy green colour of the flame confirms the presence of Ba^{2+} ions.

2. Test for Strontium ion (Sr^{2+})

(a) Solution of V group precipitate in acetic acid gives a white precipitate of strontium sulphate with ammonium sulphate solution on heating and scratching the sides of the test tube with a glass rod.

$$SrCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Sr + H_2O + CO_2$$

$$(CH_3COO)_2Sr + (NH_4)_2SO_4 \longrightarrow \underset{\substack{\text{Strontium}\\ \text{sulphate}\\ \text{(White precipitate)}}}{SrSO_4} + 2CH_3COONH_4$$

(b) **Flame test :** Perform the flame test as given in the case of Ba^{2+}. A crimson red flame confirms the presence of Sr^{2+} ions.

3. Test for Calcium ion (Ca^{2+})

(a) Solution of the fifth group precipitate in acetic acid gives a white precipitate with ammonium oxalate solution.

$$CaCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ca + H_2O + CO_2$$

$$(CH_3COO)_2Ca + \underset{\substack{\text{Ammonium}\\ \text{oxalate}}}{(NH_4)_2C_2O_4} \longrightarrow \underset{\substack{\text{Calcium oxalate}\\ \text{(White precipitate)}}}{(COO)_2Ca} + 2CH_3COONH_4$$

(b) **Flame test :** Perform the flame test as mentioned above. Calcium imparts brick red colour to the flame which looks greenish-yellow through blue glass.

(VII) Analysis of Group–VI cations

If group–V is absent then perform the test for Mg^{2+} ions as given below.

Chemistry of Confirmatory Tests of Group–VI cations

Test for Magnesium ion (Mg^{2+})

(a) If group–V is absent then the solution may contain magnesium carbonate, which is soluble in water in the presence of ammonium salts because the equilibrium is shifted towards the right hand side.

$$NH_4^+ + CO_3^{2-} \longrightarrow NH_3 + HCO_3^-$$

The concentration of carbonate ions required to produce a precipitate is not attained. When disodium hydrogenphosphate solution is added and the inner walls of the test tube are scratched with a glass rod, a white crystalline precipitate of magnesium ammonium phosphate is formed which indicates the presence of Mg^{2+} ions.

$$Mg^{2+} + Na_2HPO_4 \longrightarrow \underset{\substack{\text{Magnesium ammonium}\\ \text{phosphate (White precipitate)}}}{Mg(NH_4)PO_4} + NH_4OH + 2Na^+ + H_2O$$

Note down the observations and the inferences of the qualitative analysis in tabular form as given in the specimen record

Precautions

Fig. 1.9 : *How to smell a gas*

(a) Always use an apron, an eye protector and hand gloves while working in the chemistry laboratory.

(b) Before using any reagent or a chemical, read the label on the bottle carefully. Never use unlabelled reagent.

(c) Do not mix chemicals and reagents unnecessarily. Never taste any chemical.

(d) Be careful in smelling chemicals or vapours.

Always fan the vapours gently towards your nose (Fig. 1.9).

(e) Never add sodium metal to water or throw it in the sink or dustbin.

(f) Always pour acid into water for dilution. Never add water to acid.

(g) Be careful while heating the test tube. The test tube should never point towards yourself or towards your neighbours while heating or adding a reagent. Fig. 1.9 : How to smell a gas

(h) Be careful while dealing with the explosive compounds, inflammable substances, poisonous gases, electric appliances, glass wares, flame and the hot substances.

(i) Keep your working surroundings clean. Never throw papers and glass in the sink. Always use dustbin for this purpose.

(j) Always wash your hands after the completion of the laboratory work.

(k) Always use the reagents in minimum quantity. Use of reagents in excess, not only leads to wastage of chemicals but also causes damage to the environment.

Discussion Questions

(i) What is the difference between a qualitative and a quantitative analysis?

(ii) Can we use glass rod instead of platinum wire for performing the flame test? Explain your answer.

(iii) Why is platinum metal preferred to other metals for the flame test?

(iv) Name the anions detected with the help of dilute H_2SO_4?

(v) Why is dilute H_2SO_4 preferred over dilute HCl while testing anions?

(vi) Name the anions detected by conc. H_2SO_4.

(vii) How is sodium carbonate extract prepared ?

(viii) What is lime water and what happens on passing carbon dioxide gas through it?

(ix) Carbon dioxide gas and sulphur dioxide gas both turn lime water milky. How will you distinguish these two?

(x) How will you test the presence of carbonate ion?

(xi) What is the composition of dark brown ring which is formed at the junction of two layers in the ring test for nitrates?

(xii) Name the radical confirmed by sodium nitroprusside test.

(xiii) What is chromyl chloride test ? How do you justify that CrO_2Cl_2 is acidic in nature?

(xiv) Why do bromides and iodides not give tests similar to chromyl chloride test?

(xv) Describe the layer test for bromide and iodide ions.

(xvi) Why is silver nitrate solution stored in dark coloured bottles?

(xvii) How do you test the presence of sulphide ion?

(xviii) Why does iodine give a blue colour with starch solution?

(xix) What is Nessler's reagent?

(xx) Why is original solution for cations not prepared in conc. HNO_3 or H_2SO_4?

(xxi) Why cannot conc. HCl be used as a group reagent in place of dil. HCl for the precipitation of Ist group cations?

(xxii) How can one prevent the precipitation of Group–IV radicals, with the second group radicals?

(xxiii) Why is it essential to boil off H_2S gas before precipitation of radicals of group–III?

(xxiv) Why is heating with conc. nitric acid done before precipitation of group–III?

(xxv) Can we use ammonium sulphate instead of ammonium chloride in group–III?

(xxvi) Why is NH_4OH added before $(NH_4)_2CO_3$ solution while precipitating group–V cations?

(xxvii) Why do we sometimes get a white precipitate in group–VI even if the salt does not contain Mg^{2+} radical?

(xxviii) What is aqua regia?

(xxix) Name a cation, which is not obtained from a metal.

(xxx) How can you test the presence of ammonium ion?

(xxxi) Why are the group–V radicals tested in the order Ba^{2+}, Sr^{2+} and Ca^{2+} ?

(xxxii) Why does conc. HNO_3 kept in a bottle turn yellow in colour?

(xxxiii) Why should the solution be concentrated before proceeding to group–V?

(xxxiv) Why is the reagent bottle containing sodium hydroxide solution never stoppered?

(xxxv) What do you understand by the term common ion effect?

(xxxvi) Why is zinc sulphide not precipitated in group–II?

SPECIMEN RECORD OF SALT ANALYSIS

Aim

To analyse the given salt for one anion and one cation present in it.

Sl. No.	Experiment	Observation	Inference
1.	Noted the colour of the given salt.	White	Cu^{2+}, Fe^{2+}, Ni^{2+}, Co^{2+}, Mn^{2+} are absent.
2.	Noted the smell of the salt.	No specific smell.	S^{2-}, SO_3^{2-}, CH_3COO^- may be absent.
3.	Heated 0.5 g of the salt in a dry test tube and noted the colour of the gas evolved and change in the colour of the residue on heating and cooling.	(i) No gas was evolved. (ii) No particular change in colour of the residue is observed when heated and when cooled.	(i) CO_3^{2-} may be present, NO_3^-, NO_2^-, Br^- may be absent. (ii) Zn^{2+} may be absent.
4.	Prepared a paste of the salt with conc. HCl and performed the flame test.	No distinct colour of the flame seen.	Ca^{2+}, Sr^{2+}, Ba^{2+} Cu^{2+} may be absent.
5.	Borax bead test was not performed as the salt was white in colour.	—	—
6.	Treated 0.1 g of salt with 1 mL dil.H_2SO_4 and warmed.	No effervescence and evolution of vapours.	CO_3^{2-}, SO_3^{2-}, S^{2-}, NO_2^-, CH_3COO^- absent.
7.	Heated 0.1 g of salt with 1 mL conc. H_2SO_4.	No gas evolved.	Cl^-, Br^-, I^-, NO_3^-, $C_2O_4^{2-}$ are absent.
8.	Acidified 1mL of aqueous salt solution with conc. HNO_3. Warmed the contents and then added 4-5 drops of ammonium molybdate solution.	No yellow precipitate	PO_4^{3-} absent.

Sl. No.	Experiment	Observation	Inference
9.	Acidified water extract of the salt with dil. HCl and then added 2mL of $BaCl_2$ solution.	A white ppt. is obtained which is insoluble in conc. HNO_3 and conc. HCl.	SO_4^{2-} present.
10.	Heated 0.1 g of salt with 2 mL NaOH solution.	Ammonia gas is not evolved.	NH_4^+ absent.
11.	Attempted to prepare original solution of the salt by dissolving 1g of it in 20 mL water.	Clear solution formed	Water soluble salt is present.
12.	To a small part of the above salt solution added 2 mL of dil. HCl.	No white precipitate formed.	Group–I absent.
13.	Passed H_2S gas through one portion of the solution of step 12.	No precipitate formed.	Group–II absent.
14.	Since salt is white, heating with conc. HNO_3 is not required. Added about 0.2 g of solid ammonium chloride and then added excess of ammonium hydroxide to the solution of step 12.	No precipitate formed.	Group–III absent.
15.	Passed H_2S gas through the above solution.	No precipitate formed.	Group–IV absent.
16.	Added excess of ammonium hydroxide solution to the original solution and then added 0.5 g of ammonium carbonate.	No precipitate formed.	Group–V absent.
17.	To the original solution of salt added ammonium hydroxide solution, followed by disodium hydrogen phosphate solution. Heated and scratched the sides of the test tube.	White precipitate	Mg^{2+} confirmed.

Result

the given salt contains

Anion : SO_4^{2-}

Cation : Mg^{2+}

EXERCISE # I

ANIONS : Class A (Subgroup - I)

1. The colour developed, when sodium sulphide is added to sodium nitroprusside is:

(A) Purple (B) yellow (C) red (D) black

SA0001

2. When a neutral or slightly alkaline solution of thiosulphate is treated with the $[Ni(en)_3](NO_3)_2$ complex, then

(A) Green precipitate is obtained (B) Brown precipitate is obtained
(C) Violet precipitate is obtained (D) Yellow precipitate is obtained

SA0002

3. When CH_3COONa heated with solid As_2O_3 then compound X is formed. The smell of compound X is -

(A) Pungent smell (B) Rotten Fish smell (C) Nauseating smell (D) Rotten egg smell

SA0003

4. NO_2^- ion can be destroyed by -

(A) Sulphamic acid (B) Thiourea (C) Urea (D) All of these

SA0004

5. Solutions of sodium azide(NaN_3) and iodine (as KI_3) do not react but on addition of a trace of 'X' ion, which acts as a catalyst there is an immediate vigorous evolution of nitrogen. Then 'X' may be:

(A) $S_2O_3^{2-}$ (B) S^{2-} (C) $\overset{\ominus}{S}CN$ (D) All are correct.

SA0005

6. When $AgNO_3$ react with 'X' ion then initially no visible change occurs due to formation of water soluble complex.Then ion 'X' may be:

(A) SO_3^{2-} (B) $S_2O_3^{2-}$ (C) S^{2-} (D) CO_3^{2-}

SA0006

7. Match the column

Column-I	**Column-II**
(A) S^{2-}	(P) Produces white ppt. with excess $AgNO_3$
(B) HSO_3^-	(Q) Evolves gas with dil. HCl which turns lime water milky
(C) SO_3^{2-}	(R) Evolves gas with dil.H_2SO_4 which does **not** turn Baryta water milky
(D) $S_2O_3^{2-}$	(S) Produces ppt. with $Pb(OAc)_2$ solution.
	(T) Produces white ppt with $BaCl_2$ solution.

SA0007

8. Find the number of acidic radical(s) which can form coloured gas when treated with **dil.** H_2SO_4.
CO_3^{2-}, NO_2^-, Br^-, I^-, SO_3^{2-}

SA0008

Class A (Subgroup - II)

9. Chromyl chloride test is given by -

(A) CH_3Cl (B) AgCl (C) Hg_2Cl_2 (D) NH_4Cl

SA0009

10. $BO_3^{3-} + \underset{\text{Conc.}}{H_2SO_4} \xrightarrow{\Delta} \underset{\text{White fumes}}{(P)}$

$BO_3^{3-} + \underset{\text{Conc.}}{H_2SO_4} + C_2H_5OH \xrightarrow{\Delta} \underset{\text{Vapours}}{(Q)}$

P & Q are respectively -

(A) H_3BO_3 , H_3BO_3
(B) $(C_2H_5)_3BO_3$, H_3BO_3
(C) $(C_2H_5)_3BO_3$, $(C_2H_5)_3BO_3$
(D) H_3BO_3, $(C_2H_5)_3BO_3$

SA0010

11. In layer test of I^- and Br^-. If reddish -brown layer comes first then -

(A) Br^- present
(B) I^- absent
(C) Both (A) and (B)
(D) None of these

SA0011

All Anions Of Class A

12. **Statement-1** : When H_2S gas is passed through Na-nitroprusside soluton it gives purple colouration

Statement-2 : H_2S is an weak acid

(A) Statement-1 is true, statement-2 is true and statement-2 is correct explanation for statement-1.

(B) Statement-1 is true, statement-2 is true and statement-2 is NOT the correct explanation for statement-1.

(C) Statement-1 is true, statement-2 is false.

(D) Statement-1 is false, statement-2 is true.

SA0012

13. When the soda extract containing thiosulphate ion treated with excess of $AgNO_3$ solution followed by boiling, then.

(A) White precipitate is formed
(B) Black precipitate is formed
(C) brown precipitate is formed
(D) No ppt precipitate is formed

SA0013

14. “Cacodyl oxide” is formed in the specific test of -

(A) Formate
(B) Oxalate
(C) Acetate
(D) Nitrate

SA0014

15. An aqueous solution of gas (X) gives the white turbidity on passing H_2S in the solution. Identify (X)

(A) NH_3
(B) SO_2
(C) CO_2
(D) None of these

SA0015

16. NO_2^- and NO_3^- can be distinguished by which of the following reagent.

(A) dil. H_2SO_4
(B) conc. H_2SO_4
(C) Devarda’s alloy + conc.NaOH
(D) None of these

SA0016

17. $[Fe(H_2O)_5NO]^{2+}$ is unstable because -

(A) It liberates NO gas on warming

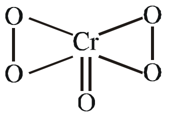
(B) It liberates NO gas on shaking

(C) The charge of central atom is +1 (relatively low enough)

(D) None of these

Class B

SA0017

18. $Cr_2O_7^{2-} + 4H_2O_2 + 2H^+ \xrightarrow[\text{Solvent}]{\text{Organic}}$ 2  $+ 5H_2O$

In above reaction amyl alcohol is recommended.

Dimethyl ether is not recommended for general use owing to its -

(A) Highly non-flammable character

(B) Highly inflammable character

(C) Highly poisonious character

(D) None of these

SA0018

19. If barium sulphate is precipitated in a solution containing potassium permanganate it is coloured pink (violet) by -

(A) Absorption of some of the permanganate

(B) Adsorption of some of the permanganate

(C) Both (A) and (B)

(D) None of these

SA0019

All Anions Of Class A & Class B

20.

List–I (Reaction)	**List–II (Product)**
(P) $KI + NO_2^- \longrightarrow$	(1) NH_3
(Q) $NH_4NO_3 \xrightarrow{\Delta}$	(2) NO
(R) $NO_2^- \xrightarrow{Zn+NaOH}$	(3) N_2
(S) $(NH_4)_2Cr_2O_7 \xrightarrow{\Delta}$	(4) N_2O

Code :

	P	**Q**	**R**	**S**
(A)	3	4	1	2
(B)	4	2	1	3
(C)	4	2	3	1
(D)	2	4	1	3

SA0020

21.

List–I (Reaction)	**List–II (Product)**
(P) $CO_3^{2-} \xrightarrow{dil.H_2SO_4}$	(1) H_2S
(Q) $S^{2-} \xrightarrow{dil.H_2SO_4}$	(2) SO_2
(R) $SO_3^{2-} \xrightarrow{dil.H_2SO_4}$	(3) CO_2
(S) $S_2O_3^{2-} \xrightarrow{dil.H_2SO_4}$	(4) $S + SO_2$

Code :

	P	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	3	4	1	2	(B)	2	1	4	3
(C)	3	1	2	4	(D)	2	4	1	3

SA0021

22.

List–I (Molecule)	**List–II (Characteristic Odour)**
(P) CO_2	(1) Rotten egg smell
(Q) SO_2	(2) Suffocating smell of burning sulphur
(R) H_2S	(3) Vineger like smell
(S) CH_3COOH	(4) Odour less

Code :

	P	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	4	2	1	3	(B)	2	4	1	3
(C)	3	1	2	4	(D)	2	4	1	3

SA0022

23.

List–I (Acidic radicals)	**List–II (Test)**
(P) NO_2^-	(1) Green flame test
(Q) BO_3^{3-}	(2) Cacodyl oxide reaction
(R) Br^-	(3) Griess Ilosvay test
(S) CH_3COO^-	(4) Layer test

Code :

	P	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	4	2	1	3	(B)	3	1	4	2
(C)	3	4	2	1	(D)	4	3	2	1

SA0023

CATIONS : DRY TEST

24. Find the number of water of crystallization in microcosmic salt -

(A) 5 (B) 4 (C) 6 (D) 10

SA0024

25. What is the colour of K^+ through cobalt/double blue glass -

(A) Lilac, (B) Violet (C) Brick red (D) Crimson red

SA0025

26. What is the colour of $CoO.Al_2O_3$ is -

(A) pink (B) Thenard blue (C) Bluish white (D) None of these

SA0026

27. The correct formula of Canary yellow ppt and it is the test of ----------- acid radical-

(A) $(NH_4)_2\ [PMo_{12}O_{40}]$ and phosphate

(B) $(NH_4)\ H\ [P(Mo_3O_{10})_4]$ and sulphate

(C) $(NH_4)_3\ [P(Mo_3O_{10})_4]$ and phosphate

(D) $Na_3\ [P(Mo_3O_{10})_4]$ and phosphate

SA0027

28. Sodium carbonate bead test generally used for ....compounds.

(A) Mn (B) Cr (C) Zn (D) Cu

SA0028

WET TEST : GROUP ZERO

29. **Statement-1 :** Test of NH_4^+ can not be done within group analysis

Statement-2 : During group analysis several times NH_4^+ - compound is added at the different steps.

(A) Statement-1 is true, statement-2 is true and statement-2 is correct explanation for statement-1

(B) Statement-1 is true, statement-2 is true and statement-2 is NOT the correct explanation for statement-1

(C) Statement-1 is false, statement-2 is true.

(D) Statement-1 is true, statement-2 is false.

SA0029

GROUP - I

30. Which of the following is not group-I cation though the chlorides of all cations are sparingly soluble in water.

(A) Ag^+ (B) Hg_2^{2+} (C) Cu^+ (D) Pb^{2+}

SA0030

GROUP - II A

31. Cu^{2+} + KCN (in excess) → soluble complex (X). How many statements are correct regarding complex (X) -

(i) the central atom has the co-ordination number of 6

(ii) the central atom has the co-ordination number of 4

(iii) the complex is sq.planar

(iv) the complex is diamagnetic

(v) the complex is paramagnetic

SA0031

32. $BiCl_3 \xrightarrow{KI}$ black ppt (M) $\xrightarrow{\text{excess KI}}$ soluble complex (N)

Find the number of moles of I^- ions involved for the formation of per mole of (N).

SA0032

GROUP - II B

33. Sn^{2+} and Sn^{4+} can be distinguished by how many of the following methods -

(i) by passing H_2S in their solution (in acidic medium)

(ii) by addition of NaOH in their solution

(iii) by addition of excess NaOH in their solution

(iv) by addition of dil. HCl in their solution

(v) by addition of $HgCl_2$ solution in their solution

SA0033

GROUP - III

34. What is the group-III reagent is generally used for group analysis.

(A) $NH_4OH + NH_4NO_3$ (B) $NH_4Cl + (NH_4)_2CO_3$

(C) $NH_4OH + (NH_4)_2SO_4$ (D) $NH_4OH + NH_4Cl$

SA0034

35. $CrCl_3$ solution + Na_2S solution $\xrightarrow{\quad}$ ppt(A)

The correct formula and colour of A are

(A) Cr_2S_3, Black (B) $Cr(OH)_3$, Green

(C) $Na[Cr(OH)_4]$, Green (D) None of these

SA0035

GROUP - IV

36. The auxiliary reagent in group-IV reagent is

(A) H_2S (B) dil.HCl (C) NaOH (D) NH_4OH

SA0036

All Group Cations

37. Which of the following cation gives ppt in two groups during group analysis.

(A) Hg^{2+} (B) Hg_2^{2+} (C) Pb^{2+} (D) Cu^{2+}

SA0037

38. Which of the following cation produces coloured ppt with Na_2SO_4 solution -

(A) Pb^{2+} solution (B) Ba^{2+} solution (C) Hg^{2+} solution (D) Ca^{2+} solution

SA0038

39. NH_4^+ and K^+ ions can be distinguished by the use of following reagent

(A) $Na_3[Co(NO_2)_6]$ (B) $Na_2[PtCl_6]$

(C) $HClO_4$ or $NaClO_4$ (D) Boiling with NaOH

SA0039

40. Which of the following sulphides is yellow in colour?

(A) CuS (B) CdS (C) ZnS (D) CoS

SA0040

MISCELLANEOUS

41.

List–I (Compound)	**List–II (Colour)**
(P) HgO	(1) Purple solution
(Q) $BaCO_3$	(2) Yellow ppt
(R) $Na_4[Fe(CN)_5NOS]$	(3) Dark brown
(S) KI_3	(4) White ppt

Code :	**P**	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	3	4	1	2	(B)	2	4	1	3
(C)	2	4	3	1	(D)	2	4	3	1

SA0041

42.

List–I (Basic Radical)	**List–II (Group)**
(P) Al^{+3}	(1) II group
(Q) Zn^{+2}	(2) V group
(R) Ba^{+2}	(3) IV group
(S) Pb^{+2}	(4) III group

Code :	**P**	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	4	2	1	3	(B)	2	4	1	3
(C)	3	1	2	4	(D)	4	3	2	1

SA0042

43.

List–I (Cations)	**List–II (Group reagent)**
(P) Co^{+2}	(1) $(NH_4)_2CO_3$ in presence of NH_4Cl
(Q) Fe^{+3}	(2) H_2S gas in acidic medium
(R) Cu^{+2}	(3) H_2S in presence of NH_4OH
(S) Ca^{+2}	(4) NH_4OH in presence of NH_4Cl

Code :	**P**	**Q**	**R**	**S**		**P**	**Q**	**R**	**S**
(A)	4	2	1	3	(B)	3	1	4	2
(C)	1	2	3	4	(D)	3	4	2	1

SA0043

EXERCISE # II

ANIONS : Class A (Subgroup - I)

1. **Statement-1:** On passing CO_2 gas through lime water, the solution turns milky.

because

Statement-2: Acid-Base (neutralisation) reaction takes place.

(A) Statement-1 is true, statement-2 is true and statement-2 is correct explanation for statement-1.

(B) Statement-1 is true, statement-2 is true and statement-2 is NOT the correct explanation for statement-1.

(C) Statement-1 is true, statement-2 is false.

(D) Statement-1 is false, statement-2 is true.

SA0044

2. A substance on treatment with dil. H_2SO_4 liberates a colourless gas which produces (i) turbidity with baryta water and (ii) turns acidified dichromate solution green. The reaction indicates the presence of

(A) CO_3^{2-} (B) S^{2-} (C) SO_3^{2-} (D) NO_2^{-}

SA0045

3. When $S_2O_3^{2-}$ react with solution of 'X' reagent then reaction is redox followed by precipitation then 'X' is:

(A) $FeCl_3$ solution (B) $AgNO_3$ solution

(C) $CuSO_4$ solution (D) None of these

SA0046

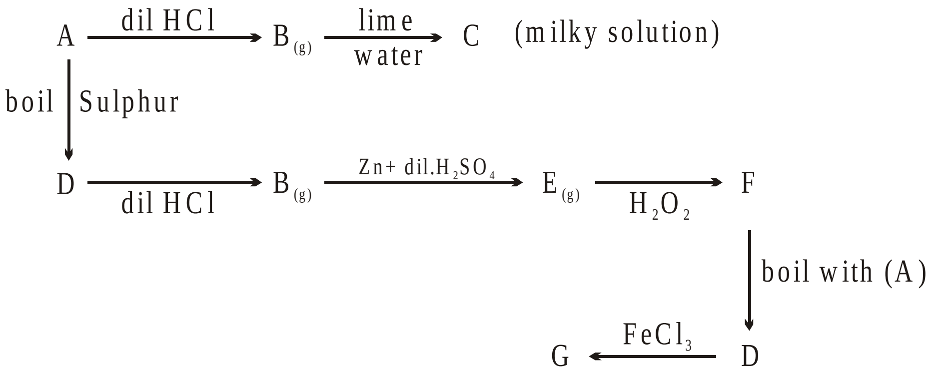
4. In the test for iodine, when I_2 is treated with sodium thiosulphate, $Na_2S_2O_3$

$$Na_2S_2O_3 + I_2 \longrightarrow NaI + ...$$

(A) $Na_2S_4O_6$ (B) Na_2SO_4 (C) Na_2S (D) Na_3ISO_4

SA0047

Paragraph for Q. 5 to Q. 7



5. Identify A

(A) CO_3^{2-} (B) SO_3^{2-} (C) $S_2O_3^{2-}$ (D) none of these

SA0048

6. When A react with $Pb(NO_3)_2$ then compound X is formed. Compound X is oxidized by atmospheric oxygen on boiling, then Y is formed what is the colour of Y

(A) yellow (B) White (C) Black (D) Green

SA0048

7. When gas E react with sodium nitroprusside in basic medium then compound Z is formed. The colour of compound Z is:

(A) Green (B) purple (C) Reddish brown (D) Black

SA0048

Class A (Subgroup - II)

8. When a mixture of solid NaCl, solid $K_2Cr_2O_7$ is heated with conc. H_2SO_4, orange red vapours are obtained. These are of the compound

(A) chromous chloride (B) chromyl chloride

(C) chromic chloride (D) chromic sulphate

SA0049

9. Which of the following will not give positive chromyl chloride test?

(A) Copper chloride, $CuCl_2$ (B) Mercuric chloride, $HgCl_2$

(C) Zinc chloride, $ZnCl_2$ (D) Aniline chloride, $C_6H_5NH_3Cl$

SA0050

10. Sodium borate on reaction with conc. H_2SO_4 and C_2H_5OH gives a compound A which burns with a green edged flame. The compound A is

(A) $H_2B_4O_7$ (B) $(C_2H_5)_2B_4O_7$ (C) H_3BO_3 (D) $(C_2H_5)_3BO_3$

SA0051

11. Nitrate is confirmed by ring test. The brown colour of the ring is due to formation of

(A) ferrous nitrite (B) nitroso ferrous sulphate

(C) ferrous nitrate (D) $FeSO_4NO_2$

SA0052

12. A salt gives violet vapours when treated with conc. H_2SO_4, it contains

(A) Cl^- (B) I^- (C) Br^- (D) NO_3^-

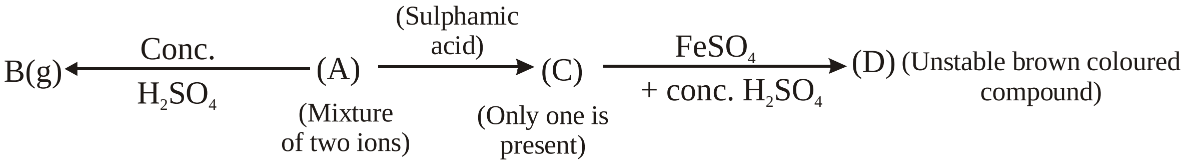
SA0053

13. Unknown salt + Al-powder + NaOH (conc.) → gas comes out which turns Nesslar's reagent brown. The salt may be -

(A) $NaNO_2$ (B) $NaNO_3$ (C) NH_4Cl (D) NH_4HCO_3

SA0054

Paragraph for Q. 14 to Q. 17



14. Identify mixture of ions (A) -

(A) NO_2^- and Br^- (B) NO_2^- and I^- (C) NO_2^- and NO_3^- (D) None of these

SA0055

15. What is oxidation state of central atom of (D)

(A) +3 (B) +2 (C) +1 (D) Zero

SA0055

16. Identify gas B-

(A) Br_2 (B) $Br_2 + NO_2$ (C) NO_2 (D) None of these

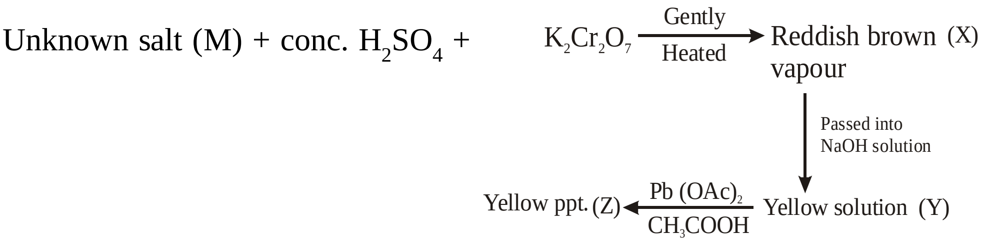
SA0055

17. What is the hybridisation of central atom of D-

(A) d^2sp^3 (B) sp^3d^2 (C) sp^3d (D) sp^3

SA0055

Paragraph for Q. 18 to Q. 20



18. The salt (M) is/are-

(A) AgCl (B) NH_4Cl + NaBr (C) NaBr (D) $Ca(ClO_4)_2$

SA0056

19. How many non axial d-orbitals are involved in hybridisation of central atom of compound (X)-

(A) 2 (B) 3 (C) 4 (D) None of these

SA0056

20. What is the formula of yellow ppt (Z)-

(A) $BaCrO_4$ (B) Na_2CrO_4 (C) Ag_2CrO_4 (D) $PbCrO_4$

SA0056

CATIONS : DRY TEST

21. In the borax bead test of Co^{2+}, the blue colour of bead is due to the formation of:

(A) B_2O_3 (B) Co_3B_2 (C) $Co(BO_2)_2$ (D) CoO

SA0057

22. Which of the following leaves no residue on heating?

(A) $Pb(NO_3)_2$ (B) NH_4NO_3 (C) $Cu(NO_3)_2$ (D) $NaNO_3$

SA0058

23. Which of the following cations is detected by the flame test?

(A) NH_4^+ (B) K^+ (C) Mg^{2+} (D) Al^{3+}

SA0059

24. Which metal salt gives a violet coloured bead in the borax bead test (oxidising flame, cold)?

(A) Fe^{2+} (B) Ni^{2+} (C) Co^{2+} (D) Mn^{2+}

SA0060

25. The compound formed in the borax bead test of Cu^{2+} ion in oxidising flame is:

(A) Cu (B) $CuBO_2$ (C) $Cu(BO_2)_2$ (D) None of these

SA0061

26. In microcosmic salt bead test Co^{2+} produce blue bead due to the formation of -

(A) $Cu(BO_2)_2$ (B) $NaCoPO_4$ (C) $Co_2(PO_4)(BO_2)$ (D) $NaPO_3$

SA0062

Paragraph for Q. 27 to Q. 30

$$\underset{\text{(Hydrated salt)}}{(A)} \xrightarrow[\text{heated}]{\text{Strongly}} \underbrace{B + C}_{\substack{\text{transparent}\\ \text{glassy bead}}}$$

$$H_3BO_3 \xrightarrow[\text{heated}]{\text{Strongly}} C + D$$

27. Identify C-

(A) $(BN)_X$ (B) $NaPO_3$ (C) B_2O_3 (D) $Mg(NH_4)PO_4$

SA0063

28. Find the number of water of crystallizations in (A)-

(A) 4 (B) 5 (C) 10 (D) 24

SA0063

29. How many X–O–X linkages are present in structure of A (X = central atom)-

(A) 4 (B) 3 (C) 5 (D) 2

SA0063

30. Find the number of tetrahedral and trigonal planar units in structure of A -

(A) 2,1 (B) 2,2 (C) 2,4 (D) 5,2

SA0063

WET TEST : GROUP - I

31. Mercurous ion is represented as:

(A) Hg_2^{2+} (B) Hg^{2+} (C) $Hg + Hg^{2+}$ (D) Hg_2^+

SA0064

32. A white sodium salt dissolves readily in water to give a solution which is neutral to litmus. When silver nitrate solution is added to the solution, a white precipitate is obtained which does not dissolve in dil. HNO_3. The anion could be:

(A) CO_3^{2-} (B) Cl^- (C) SO_4^{2-} (D) S^{2-}

SA0065

33. An aqueous solution of a substance gives a white ppt. on treatment with dil. HCl, which dissolves on heating. When hydrogen sulphide is passed through the hot acidic solution, a black ppt. is obtained. The substance is a

(A) Hg^{2+} salt (B) Cu^{2+} salt (C) Ag^+ salt (D) Pb^{2+} salt

SA0066

34. A white ppt obtained in a analysis of a mixture becomes black on treatment with NH_4OH. It may be

(A) $PbCl_2$ (B) AgCl (C) $HgCl_2$ (D) Hg_2Cl_2

SA0067

GROUP - II

35. When bismuth chloride is poured into a large volume of water the white precipitate produced is

(A) $Bi(OH)_3$ (B) Bi_2O_3 (C) BiOCl (D) Bi_2OCl_3

SA0068

36. $CuSO_4$ decolourises on addition of excess KCN, the product is

(A) $[Cu(CN)_4]^{2-}$.

(B) Cu^{2+} get reduced to form $[Cu(CN)_4]^{3-}$

(C) $Cu(CN)_2$

(D) CuCN

SA0069

37. When H_2S gas is passed through the HCl containing aqueous solution of $CuCl_2$, $HgCl_2$, $BiCl_3$ and $CoCl_2$, it does not precipitate out:

(A) CuS (B) HgS (C) Bi_2S_3 (D) CoS

SA0070

38. Which of the following is soluble in yellow ammonium sulphide?

(A) CuS (B) CdS (C) SnS (D) PbS

SA0071

39. When excess of $SnCl_2$ is added to a solution of $HgCl_2$, a white ppt turning grey is obtained. The grey colour is due to the formation of

(A) Hg_2Cl_2 (B) $SnCl_4$ (C) Sn (D) Hg

SA0072

40. On passing H_2S gas in II group sometimes the solution turns milky. It indicates the presence of

(A) oxidising agent (B) acidic salt (C) s-block cation (D) reducing agent.

SA0073

41. Which fo the following yellow coloured sulphide is insouble in yellow ammonium sulphide.

(A) SnS_2 (B) As_2S_5 (C) CdS (D) Bi_2S_3

SA0074

42. Type of sulphide ppt may be obtained in the group-II ppt during group analysis.

(A) M_2S_3 (B) M_2S (C) MS (D) MS_2

SA0075

43. The metal ion which is precipitated when H_2S is passed with HCl:

(A) Zn^{2+} (B) Ni^{2+} (C) Cd^{2+} (D) Mn^{2+}

SA0076

GROUP - III

44. In the precipitation of the iron group in qualitative analysis, ammonium chloride is added before adding ammonium hydroxide to

(A) decrease concentration of OH^- ions. (B) prevent interference by phosphate ions.

(C) increase concentration of Cl^- ions. (D) increase concentration of NH_4^+ ions.

SA0077

45. If reddish brown ppt (only) is obtained in group-III during group analysis, then oxidation state of Fe in the original sample may be

(A) +2 (B) +3 (C) +2 and +3 both (D) Neither +2 nor +3

SA0078

46. If NH_4Cl is not added to the group-III reagent which of the following ppt could be obtained

(A) $Cr(OH)_2$ (B) $Fe(OH)_3$ (C) $Mn(OH)_2$ (D) $Mg(OH)_2$

SA0079

47. In which of the following cases blue ppt is obtained

(A) $Fe^{2+} + [Fe(CN)_6]^{3-} \longrightarrow$ (B) $Fe^{2+} + [Fe(CN)_6]^{4-} \longrightarrow$

(C) $Fe^{3+} + [Fe(CN)_6]^{4-} \longrightarrow$ (D) $Fe^{3+} + [Fe(CN)_6]^{3-} \xrightarrow{SnCl_2}$

SA0080

48. What are the following steps are to be done before adding group-III reagent into the group-II filtrate.

(A) Group-II filtrate is to be evapourated to dryness

(B) Group-II filtrate is to be boiled of first

(C) After boiling 2-3 drops of dil.H_2SO_4 is added and boiled again.

(D) After boiling 2-3 drops of HNO_3 is added and boiled again.

SA0081

49. A pale green crystalline metal salt of M dissolves freely in water. On standing it gives a brown ppt on addition of aqueous NaOH. The metal salt solution also gives a black ppt on bubbling H_2S in basic medium. An aqueous solution of the metal salt decolourizes the pink colour of the permanganate solution. The metal in the metal salt solution is

(A) copper (B) aluminium (C) lead (D) iron

SA0082

50. Which of the following compound on reaction with NaOH and Na_2O_2 gives yellow colour?

(A) $Cr(OH)_3$ (B) $Zn(OH)_2$ (C) $Al(OH)_3$ (D) None of these

SA0083

GROUP - IV

51. Colour of nickel chloride solution is

(A) pink (B) black (C) colourless (D) green

SA0084

52. Dimethyl glyoxime in a suitable solvent was refluxed for 10 minutes with pure pieces of nickel sheet, it will result in

(A) Red ppt (B) Blue ppt. (C) Yellow ppt. (D) No ppt.

SA0085

53. Which one of the following does not produce metallic sulphide with H_2S?

(A) $ZnCl_2$(Neutral sol^n) (B) $CdCl_{2(aq)}$ (C) $CoCl_{2(aq)}$ (D) $CuCl_{2(aq)}$

SA0086

54. Which is not dissolved by dil HCl?

(A) ZnS (B) MnS (C) $BaSO_3$ (D) $BaSO_4$

SA0087

GROUP - V

55. In III group, NH_4Cl is added to decrease concentation of hydroxide ion by NH_4OH. We do not add $(NH_4)_2SO_4$ along with NH_4OH because -

(A) $(NH_4)_2SO_4$ is insoluble in water (B) It precipitate other insoluble sulphates

(C) It is weak electrolyte (D) None of these

SA0088

GROUP - VI

56. A metal is burnt in air and the ash on moistening smells of ammonia. The metal is

(A) Na (B) Fe (C) Mg (D) Al

SA0089

57. A metal 'X' on heating in nitrogen gas gives 'Y' . 'Y' on treatment with H_2O gives a colourless gas which when passed through $CuSO_4$ solution gives a blue colour Y is:

(A) $Mg(NO_3)_2$ (B) Mg_3N_2 (C) NH_3 (D) MgO

SA0090

MISCELLANEOUS

58. Na_2HPO_4 + Reagent ‘M’ $\rightarrow$ white ppt. The reagent ‘M’ is -

(A) $BaCl_2$ solution (B) $AlCl_3$ solution (C) $MnSO_4$ solution (D) $FeCl_3$ solution

SA0091

59. A white solid is first heated with dil H_2SO_4 and then with conc. H_2SO_4. No action was observed in either case. The solid salt contains

(A) sulphide (B) sulphite (C) thiosulphate (D) sulphate

SA0092

60. A mixture of chlorides of copper, cadmium, chromium, iron and aluminium was dissolved in water acidified with HCl and hydrogen sulphide gas was passed for sufficient time. It was filtered, boiled and a few drops of nitric acid were added while boiling. To this solution ammonium chloride and sodium hydroxide were added in excess and filtered. The filtrate shall give test for

(A) sodium and iron ion (B) sodium, chromium and aluminium ion

(C) aluminium and iron ion (D) sodium, iron, cadmium and aluminium ion

SA0093

61. In Nessler's reagent, the ion present is:

(A) HgI^{2-} (B) HgI_4^{2-} (C) Hg^+ (D) Hg^{2+}

SA0094

62. The cations present in slightly acidic solution are Fe^{3+}, Zn^{2+} and Cu^{2+}. The reagent which when added in excess to this solution would identify and separte Fe^{3+} in one step is:

(A) 2 M HCl (B) 6 M NH_3 (C) 6 M NaOH (D) H_2S gas

SA0095

63. In the separation of Cu^{2+} and Cd^{2+} in 2nd group qualitative analysis of cation, tetrammine copper (II) sulphate and tetrammine cadmium (II) sulphate react with KCN to form the corresponding cyano complexes. Which one of the following pairs of the complexes and their relative stability enables the separation of Cu^{2+} and Cd^{2+}?

(A) $K_3[Cu(CN)_4]$ more stable and $K_2[Cd(CN)_4]$ less stable.

(B) $K_2[Cu(CN)_4]$ less stable and $K_2[Cd(CN)_4]$ more stable.

(C) $K_2[Cu(CN)_4]$ more stable and $K_2[Cd(CN)_4]$ less stable.

(D) $K_3[Cu(CN)_4]$ less stable and $K_2[Cd(CN)_4]$ more stable.

SA0096

64. Which one has the minimum solubility product?

(A) AgCl (B) $AlCl_3$ (C) $BaCl_2$ (D) NH_4Cl

SA0097

65. Which of the following sulphate is insoluble in water?

(A) $CuSO_4$ (B) $CdSO_4$ (C) $PbSO_4$ (D) $Bi_2(SO_4)_3$

SA0098

66. Which of the following gives blood red colour with KSCN?

(A) Cu^{2+} (B) Fe^{3+} (C) Al^{3+} (D) Zn^{2+}

SA0099

67. Which one of the following metal sulphide has maximum solubility in water?

(A) HgS, $K_{sp} = 10^{-54}$ (B) CdS, $K_{sp} = 10^{-30}$

(C) FeS, $K_{sp} = 10^{-20}$ (D) ZnS, $K_{sp} = 10^{-22}$

SA0100

68. Identify the correct order of solubility of Na_2S, CuS and ZnS in aqueous medium is:

(A) CuS > ZnS > Na_2S (B) ZnS > Na_2S >CuS

(C) Na_2S > CuS > ZnS (D) Na_2S > ZnS > CuS

SA0101

69. Match the column -

Column-I **(Element)**	**Column-II** **(Correct characteristics)**
(A) Ba	(P) cation in solution produces brick red ppt. with CrO_4^{2-}
(B) Pb	(Q) cation in solution produces yellow ppt .with CrO_4^{2-}
(C) Ag	(R) corresponding salt produces apple green colour in the flame test
(D) Ca	(S) corresponding salt produces brick red colour in the flame test
	(T) cation in solution produces no ppt. with CrO_4^{2-} ion

SA0102

70.

Column-I **Cation in solution**	**Column-I** **Correct characteristics when no where excess reagent is used**
(A) Ag^+ and Pb^{2+}	(P) can be distinguished by Na_2HPO_4 solution
(B) Zn^{2+} and Mg^{2+}	(Q) can be distinguished by dil.HCl
(C) Pb^{2+} and Hg_2^{2+}	(R) can be distinguished by KI solution
(D) Ag^+ and Fe^{3+}	(S) can not be distinguished by NH_4OH solution

SA0103

The following column 1, 2, 3 represent the various tests carried out for identification of various group basic radicals, using various reagents and nature of reaction/properties of products observed. Answer the questions that follow

Column-1 - Cations/Basic Radical

Column-2 - Excess Reagent used with cation

Column-3 - Nature of Reaction/Properties of product formed

Column - 1 Cations	**Column - 2 Excess Reagent used with cation**	**Column - 3 Nature of Reaction/ Properties of product formed**
(I) Cu^{2+}	(i) KI (< 6M)	(P) Reduction of cation occurs
(II) Fe^{3+}	(ii) $K_4[Fe(CN)_6]$	(Q) Coloured complex formation
(III) Pb^{2+}	(iii) KCN	(R) Precipitation occurs
(IV) Ni^{2+}	(iv) NH_4OH	(S) Diamagnetic & square planar complex formation

71. For a **group-II** basic radical, which is the only **INCORRECT** combination?

(A) (I), (i), (P) (B) (IV), (iii), (S) (C) (III), (iv), (R) (D) (III), (iii), (R)

SA0104

72. For a **group-IV** basic radical, which is the only **CORRECT** combination?

(A) (I), (iv), (S) (B) (IV), (iii), (P) (C) (II), (iv), (Q) (D) (IV), (iv), (Q)

SA0104

73. Which combination has a entirely different colour from others?

(A) (IV), (iv), (Q) (B) (I), (iv), (Q) (C) (II), (iii), (Q) (D) (II), (ii), (Q)

SA0104

74. How many of the following gives green ppt.

(i) $CrCl_3 + NaOH \rightarrow$ (ii) $CrCl_3$ + excess $NaOH \rightarrow$

(iii) $NiCl_2$ + excess $NaOH \rightarrow$ (iv) $NiCl_2$ + excess $NH_4OH \rightarrow$

(v) $Hg_2^{2+} + KI \rightarrow$

SA0104

75. Find the no. of cation which gives white ppt with $K_4[Fe(CN)_6]$

Sr^{2+} Ca^{2+}, Zn^{2+}, Fe^{3+} , Cu^{2+}

SA0104

EXERCISE # JEE MAINS

1. Which products are expected from the disproportionation of hypochlorous acid :**[AIEEE-2002]**

(1) $HClO_3$ and Cl_2O (2) $HClO_2$ and $HClO$ (3) HCl and Cl_2O (4) HCl and $HClO_3$

SA0105

2. A metal M readily forms its sulphate MSO_4 which is water soluble. It forms oxide MO which becomes inert on heating. It forms insoluble hydroxide which is soluble in NaOH. The metal M is:-

[AIEEE-2002]

(1) Mg (2) Ba (3) Ca (4) Be

SA0106

3. Which statement is correct :- **[AIEEE-2003]**

(1) Fe^{3+} ions give deep green precipitate with $K_4[Fe(CN)_6]$

(2) On heating K^+, Ca^{2+} and HCO_3^- ions, we get a precipitate of $K_2[Ca(CO_3)_2]$

(3) Manganese salts give a violet borax bead test in reducing flame

(4) From a mixed precipitate of AgCl and AgI ammonia solution dissolves only AgCl

SA0107

4. What would happen when a solution of potassium chromate is treated with an excess of dilute nitric acid - **[AIEEE-2003]**

(1) Cr^{3+} and $Cr_2O_7^{2-}$ are formed (2) $Cr_2O_7^{2-}$ and H_2O are formed

(3) $Cr_2O_7^{2-}$ is reduced to +3 state of Cr (4) $Cr_2O_7^{2-}$ is oxidised to +7 state of Cr

SA0108

5. Ammonia forms the complex ion $[Cu(NH_3)_4]^{2+}$ with copper ions in alkaline solutions but not in acidic solution. What is the reason for it :- **[AIEEE-2003]**

(1) In acidic solutions hydration protects copper ions

(2) In acidic solutions protons coordinate with ammonia molecules forming NH_4^+ ions and NH_3 molecules are not available

(3) In alkaline solutions insoluble $Cu(OH)_2$ is precipitated which is soluble in excess of any alkali

(4) Copper hydroxide is an amphoteric substance

SA0109

6. Excess of KI reacts with $CuSO_4$ solution and then $Na_2S_2O_3$ solution is added to it. Which of the statements is incorrect for this reaction : **[AIEEE-2004]**

(1) Evolved I_2 is reduced (2) CuI_2 is formed

(3) $Na_2S_2O_3$ is oxidised (4) Cu_2I_2 is formed

SA0110

7. Calomel on reaction with NH_4OH gives **[AIEEE-2004]**

(1) $HgNH_2Cl$ (2) $NH_2–Hg–Hg–Cl$ (3) Hg_2O (4) HgO

SA0111

8. One mole of magnesium nitride on reaction with excess of water gives :- **[AIEEE-2004]**

(1) Two mole of HNO_3 (2) Two mole of NH_3

(3) 1 mole of NH_3 (4) 1 mole of HNO_3

SA0112

9. The products obtained on heating $LiNO_3$ will be :- **[AIEEE-2011]**

(1) $LiNO_2 + O_2$ (2) $Li_2O + NO_2 + O_2$

(3) $Li_3N + O_2$ (4) $Li_2O + NO + O_2$

SA0113

10. What is the best description of the change that occurs when $Na_2O(s)$ is dissolved in water ?

(1) Oxidation number of sodium decreases **[AIEEE-2011]**

(2) Oxide ion accepts sharing in a pair of electrons

(3) Oxide ion donates a pair of electrons

(4) Oxidation number of oxygen increases

SA0114

11. Which of the following on thermal-decomposition yields a basic as well as an acidic oxide ? **[AIEEE-2012]**

(1) NH_4NO_3 (2) $NaNO_3$ (3) $KClO_3$ (4) $CaCO_3$

SA0115

12. The correct statement for the molecule, CsI_3, is: **[JEE(Main)-2014]**

(1) it contains Cs^{3+} and $I^–$ ions (2) it contains Cs^+, $I^–$ and lattice I_2 molecule

(3) it is a covalent molecule (4) it contains Cs^+ and I_3^- ions

SA0116

13. A metal M on heating in nitrogen gas gives Y. Y on treatment with H_2O gives a colourless gas which when passed through $CuSO_4$ solution gives a blue colour, Y is :- **[JEE(Main)-2012 online_P-4]**

(1) NH_3 (2) MgO (3) Mg_3N_2 (4) $Mg(NO_3)_2$

SA0117

14. Potassium dichromate when heated with concentrated sulphuric acid and a soluble chloride, gives brown - red vapours of: **[JEE(Main)-2013 online_P-1]**

(1) CrO_3 (2) Cr_2O_3 (3) $CrCl_3$ (4) CrO_2Cl_2

SA0118

15. Sodium Carbonate cannot be used in place of $(NH_4)_2CO_3$ for the identification of Ca^{2+} , Ba^{2+} and Sr^{2+} ions (in group V) during mixture analysis because : **[JEE(Main)-2013 online_P-1]**

(1) Sodium ions will react with acid radicals

(2) Concentration of $CO_3^{2–}$ ions is very low

(3) Mg^{2+} ions will also be precipitated

(4) Na^+ ions will interfere with the detection of Ca^{2+}, Ba^{2+}, Sr^{2+} ions

SA0119

16. Which of the following statements is incorrect? **[JEE(Main)-2013 online_P-2]**

(1) Fe^{2+} ion also gives blood red colour with SCN^- ion

(2) Cupric ion reacts with excess of ammonia solution to give deep blue colour of $[Cu(NH_3)_4]^{2+}$ ion.

(3) Fe^{3+} ion gives blood red colour with SCN^- ion.

(4) On passing H_2S into Na_2ZnO_2 solution, a white ppt of ZnS is formed.

SA0120

17. Identify incorrect statement **[JEE(Main)-2013 online_P-3]**

(1) Copper (I) compounds are colourless except where colour results from charge transfer

(2) Copper (I) compounds are diamagnetic

(3) Cu_2S is black

(4) Cu_2O is colourless

SA0121

18. Which one of the following cannot function as an oxidising agent ?**[JEE(Main)-2013 online_P-4]**

(1) $NO_3^-(aq)$ (2) I^- (3) $Cr_2O_7^{2-}$ (4) $S_{(S)}$

SA0122

19. Which of the following statements about Na_2O_2 is **not** correct ? **[JEE(Main)-2014 online_P-2]**

(1) Na_2O_2 oxidises Cr^{3+} to CrO_4^{2-} in acid medium

(2) It is diamagnetic in nature

(3) It is the super oxide of sodium

(4) It is a derivative of H_2O_2

SA0123

20. Consider the following equilibrium **[JEE(Main)-2014 online_P-2]**

$$AgCl\downarrow + 2NH_3 \rightleftharpoons [Ag(NH_3)_2]^+ + Cl^-$$

White precipitate of AgCl appears on adding which of the following?

(1) NH_3 (2) Aqueous NaCl (3) Aqueous NH_4Cl (4) Aqueous HNO_3

SA0124

21. Consider the reaction **[JEE(Main)-2014 online_P-4]**

$$H_2SO_{3(aq)} + Sn^{4+}_{(aq)} + H_2O_{(l)} \rightarrow Sn^{2+}_{(aq)} + HSO^-_{4(aq)} + 3H^+_{(aq)}$$

Which of the following statements is correct?

(1) H_2SO_3 is the reducing agent because it undergoes oxidation

(2) H_2SO_3 is the reducing agent because it undergoes reduction

(3) Sn^{4+} is the reducing agent because it undergoes oxidation

(4) Sn^{4+} is the oxidizing agent because it undergoes oxidation

SA0125

22. Amongst the following, identify the species with an atom in +6 oxidation state :

[JEE(Main)-2014 online_P-4]

(1) $[MnO_4]^-$ (2) $[Cr(CN)_6]^{3-}$ (3) Cr_2O_3 (4) CrO_2Cl_2

SA0126

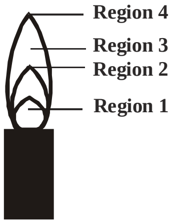
23. The hottest region of Bunsen flame shown in the figure below is : **[JEE(Main)-2016]**

(1) region 4

(2) region 1

(3) region 2

(4) region 3



SA0127

24. Sodium extract is heated with concentrated HNO_3 before testing for halogens because :

(1) S^{2-} and CN^-, if present are decomposed by conc. HNO_3 and hence do not interfere in the test.

(2) Ag reacts faster with halides in acidic medium **[JEE(Main)-2016 online]**

(3) Ag_2S and AgCN are soluble in acidic medium.

(4) Silver halides are totally insoluble in nitric acid.

SA0128

25. In the following reactions, ZnO is respectively acting as a/an :

(a) $ZnO + Na_2O \rightarrow Na_2ZnO_2$ (b) $ZnO + CO_2 \rightarrow ZnCO_3$ **[JEE(Main)-2017 off line]**

(1) base and acid (2) base and base (3) acid and acid (4) acid and base

SA0129

26. The products obtained when chlorine gas reacts with cold and dilute aqueous NaOH are :-

(1) ClO^- and ClO_3^- (2) ClO_2^- and ClO_3^- **[JEE(Main)-2017 off line]**

(3) Cl^- and ClO^- (4) Cl^- and ClO_2^-

SA0130

27. Sodium salt of an organic acid 'X' produces effervescence with conc. H_2SO_4. 'X' reacts with the acidified aqueous $CaCl_2$ solution to give a white precipitate which decolourises acidic solution of $KMnO_4$. 'X' is :- **[JEE(Main)-2017 off line]**

(1) C_6H_5COONa (2) HCOONa (3) CH_3COONa (4) $Na_2C_2O_4$

SA0131

28. A solution containing a group-IV cation gives a precipitate on passing H_2S. A solution of this precipitate in dil. HCl produces a white precipitate with NaOH solution and bluish-white precipitate with basic potassium ferrocyanide. The cation is : **[JEE(Main)-2017 on line]**

(1) Mn^{2+} (2) Zn^{2+} (3) Ni^{2+} (4) Co^{2+}

SA0132

29. Which of the following ions does **not** liberate hydrogen gas on reaction with dilute acids?

(1) Ti^{2+} (2) Cr^{2+} **[JEE(Main)-2017 on line]**

(3) Mn^{2+} (4) V^{2+}

SA0133

30. When metal 'M' is treated with NaOH, a white gelatinous precipitate 'X' is obtained, which is soluble in excess of NaOH. Compound 'X' when heated strongly gives an oxide which is used in chromatography as an adsorbent. The metal 'M' is **[JEE(Main)-2018 off line]**

(1) Ca (2) Al (3) Fe (4) Zn

SA0134

31. A white sodium salt dissolves readily in water to give a solution which is neutral to litmus. When silver nitrate solution is added to the aforementioned solution, a white precipitate is obtained which does not dissolve in dil. nitric acid. The anion is : **[JEE(Main)-2018 on line]**

(1) S^{2-} (2) SO_4^{2-} (3) CO_3^{2-} (4) Cl^{-}

SA0135

32. The incorrect statement is :- **[JEE(Main)-2018 on line]**

(1) Ferric ion gives blood red colour with potassium thiocyanate.

(2) Cu^{2+} and Ni^{2+} ions give black precipitate with H_2S in presence of HCl solution.

(3) Cu^{2+} salts give red coloured borax bead test in reducing flame.

(4) Cu^{2+} ion gives chocolate coloured precipitate with potassium ferrocyanide solution.

SA0136

33. When XO_2 is fused with an alkali metal hydroxide in presence of an oxidizing agent such as KNO_3; a dark green product is formed which disproportionates in acidic solution to afford a dark purple solution. X is : **[JEE(Main)-2018 on line]**

(1) Ti (2) Cr (3) V (4) Mn

SA0137

EXERCISE # J-ADVANCED

1. Which of the following statement(s) is (are) correct with reference to the ferrous and ferric ions:

(A) Fe^{3+} gives brown colour with potassium ferricyanide **[JEE 1998]**

(B) Fe^{2+} gives blue precipitate with potassium ferricyanide

(C) Fe^{3+} give red colour with potassium thiocyanate

(D) Fe^{2+} gives brown colour with ammonium thiocyanate

SA0138

2. Which of the following statement(s) is /are correct. When a mixture of NaCl and $K_2Cr_2O_7$ is gently warmed with conc. H_2SO_4? **[JEE 1998]**

(A) A deep red vapours is evolved.

(B) The vapours when passed into NaOH solution gives a yellow solution of Na_2CrO_4

(C) Chlorine gas is evolved

(D) Chromyl chloride is formed.

SA0139

3. An aqueous solution of a substance gives a white precipitate on treatment with dilute hydrochloric acid, which dissolves on heating. When hydrogen sulphide is passed through the hot acidic solution, a black precipitate is obtained. The substance is a : **[JEE 2000]**

(A) Hg_2^+ salt (B) Cr^{2+} salt (C) Ag^+ salt (D) Pb^{2+} salt

SA0140

4. A gas 'X' is passed through water to form a saturated solution. The aqueous solution on treatment with silver nitrate gives a white precipitate. The saturated aqueous solution also dissolves magnesium ribbon with evolution of a colourless gas 'Y'. Identify 'X' and 'Y': **[JEE 2002(Mains)]**

(A) $X = CO_2$, $Y = Cl_2$ (B) $X = Cl_2$, $Y = CO_2$

(C) $X = Cl_2$, $Y = H_2$ (D) $X = H_2$, $Y = Cl_2$

SA0141

5. $[X] + H_2SO_4 \rightarrow [Y]$ a colourless gas with irritating smell **[JEE 2003]**

$[Y] + K_2Cr_2O_7 + H_2SO_4 \longrightarrow$ green solution

[X] and [Y] are:

(A) SO_3^{2-}, SO_2 (B) Cl^-, HCl (C) S^{2-}, H_2S (D) CO_3^{2-}, CO_2

SA0142

6. A sodium salt of an unknown anion when treated with $MgCl_2$ give white precipitate only on boiling. The anion is: **[JEE 2004]**

(A) SO_4^{2-} (B) HCO_3^- (C) CO_3^{2-} (D) NO_3^-

SA0143

7. $(NH_4)_2Cr_2O_7$ on heating gives a gas which is also given by: **[JEE 2004]**

(A) heating NH_4NO_2 (B) heating NH_4NO_3

(C) $Mg_3N_2 + H_2O$ (D) $NaNO_2 + H_2O_2$

SA0144

8. A metal nitrate reacts with KI to give a black precipitate which on addition of excess of KI convert into orange colour solution. The cation of metal nitrate is: **[JEE 2005]**

(A) Hg^{2+} (B) Bi^{3+} (C) Pb^{2+} (D) Cu^+

SA0145

9. A solution when diluted with H_2O and boiled, it gives a white precipitate. On addition of excess NH_4Cl / NH_4OH, the volume of precipitate decreases leaving behind a white gelatinous precipitate. Identify the precipitate which dissolves in NH_4OH / NH_4Cl. **[JEE 2006]**

(A) $Zn(OH)_2$ (B) $Al(OH)_3$ (C) $Mg(OH)_2$ (D) $Ca(OH)_2$

SA0146

10. $CuSO_4$ decolourises on addition of excess KCN, the product is: **[JEE 2006]**

(A) $[Cu(CN)_4]^{2-}$ (B) Cu^{2+} get reduced to form $[Cu(CN)_4]^{3-}$

(C) $Cu(CN)_2$ (D) CuCN

SA0147

11. Consider a titration of potassium dichromate solution with acidified Mohr's salt solution using diphenylamine as indicator. The number of moles of Mohr's salt required per mole of dichromate is: **[JEE 2007]**

(A) 3 (B) 4 (C) 5 (D) 6

SA0148

12. The species present in solution when CO_2 is dissolved in water are **[JEE 2007]**

(A) $CO_2, H_2CO_3, HCO_3^-, CO_3^{2-}$ (B) H_2CO_3, CO_3^{2-}

(C) CO_3^{2-}, HCO_3^- (D) CO_2, H_2CO_3

SA0149

13. Sodium fusion extract, obtained from aniline, on treatment with iron (II) sulphate and H_2SO_4 in presence of air gives a Prussian blue precipitate. The blue colour is due to the formation of :

[JEE 2007]

(A) $Fe_4[Fe(CN)_6]_3$ (B) $Fe_3[Fe(CN)_6]_2$

(C) $Fe_4[Fe(CN)_6]_2$ (D) $Fe_3[Fe(CN)_6]_3$

SA0150

14. **[JEE 2007]**

Column I	**Column II**
(A) $O_2^{-} \rightarrow O_2 + O_2^{2-}$	(P) Redox reaction
(B) $CrO_4^{2-} + H^+ \rightarrow$	(Q) One of the products has trigonal planar structure
(C) $MnO_4^{-} + NO_2^{-} + H^+ \rightarrow$	(R) Dimeric bridged tetrahedral metal ion
(D) $NO_3^{-} + H_2SO_4 + Fe^{2+} \rightarrow$	(S) Disproportionation

SA0151

15. A solution of a metal ion when treated with KI gives a red precipitate which dissolves in excess KI to give a colourless solution. Moreover, the solution of metal ion on treatment with a solution of cobalt (II) thiocyanate gives rise to a deep blue crystalline precipitate. The metal ion is

[JEE 2007]

(A) Pb^{2+} (B) Hg^{2+} (C) Cu^{2+} (D) Co^{2+}

SA0152

16. A solution of colourless salt H on boiling with excess NaOH produces a non-flammable gas. The gas evolution ceases after sometime. Upon addition of Zn dust to the same solution, the gas evolution restarts. The colourless salt(s) H is (are) **[JEE 2008]**

(A) NH_4NO_3 (B) NH_4NO_2 (C) NH_4Cl (D) $(NH_4)_2SO_4$

SA0153

Paragraph for Question Nos. 17 to 19

p-Amino-N, N-dimethylaniline is added to a strongly acidic solution of **X**. The resulting solution is treated with a few drops of aqueous solution of **Y** to yield blue coloration due to the formation of methylene blue. Treatment of the aqueous solution of **Y** with the reagent potassium hexacyanoferrate(II) leads to the formation of an intense blue precipitate. The precipitate dissolves on excess addition of the reagent. Similarly, treatment of the solution of **Y** with the solution of potassium hexacyanoferrate(III) leads to a brown coloration due to the formation of **Z**. **[JEE 2009]**

17. The compound **X** is

(A) $NaNO_3$ (B) NaCl (C) Na_2SO_4 (D) Na_2S

SA0154

18. The compound **Y** is

(A) $MgCl_2$ (B) $FeCl_2$ (C) $FeCl_3$ (D) $ZnCl_2$

SA0154

19. The compound Z is

(A) $Mg_2[Fe(CN)_6]$ (B) $Fe[Fe(CN)_6]$ (C) $Fe_4[Fe(CN)_6]_3$ (D) $K_2Zn_3[Fe(CN)_6]_2$

SA0154

20. Match each of the reactions given in Column I with the corresponding product(s) given in Column II.

[JEE 2009]

Column I	**Column II**
(A) Cu + dil. HNO_3	(P) NO
(B) Cu + conc. HNO_3	(Q) NO_2
(C) Zn + dil. HNO_3	(R) N_2O
(D) Zn + conc. HNO_3	(S) $Cu(NO_3)_2$
	(T) $Zn(NO_3)_2$

SA0155

21. Passing H_2S gas into a mixture of Mn^{2+}, Ni^{2+}, Cu^{2+} and Hg^{2+} ions in an acidified aqueous solution precipitates **[JEE 2011]**

(A) CuS and HgS (B) MnS and CuS (C) MnS and NiS (D) NiS and HgS

SA0156

22. Reduction of the metal centre in aqueous permanganate ion involves - **[JEE 2011]**

(A) 3 electrons in neutral medium (B) 5 electrons in neutral medium

(C) 3 electrons in weak alkaline medium (D) 5 electrons in acidic medium

SA0157

23. The equilibrium **[JEE 2011]**

$$2Cu^{I} \rightleftharpoons Cu^{o} + Cu^{II}$$

in aqueous medium at 25°C shifts towards the left in the presence of

(A) NO_3^- (B) Cl^- (C) SCN^- (D) CN^-

SA0158

Paragraph for Questions Nos. 24 to 26

When a metal rod M is dipped into an aqueous colourless concentrated solution of compound N, the solution turns light blue. Addition of aqueous NaCl to the blue solution gives a white precipitate O. Addition of aqueous NH_3 dissolves O and gives are intense blue solution. **[JEE 2011]**

24. The metal rod M is -

(A) Fe (B) Cu (C) Ni (D) Co

SA0159

25. The compound N is -

(A) $AgNO_3$ (B) $Zn(NO_3)_2$ (C) $Al(NO_3)_3$ (D) $Pb(NO_3)_2$

SA0159

26. The final solution contains -

(A) $[Pb(NH_3)_4]^{2+}$ and $[CoCl_4]^{2-}$ (B) $[Al(NH_3)_4]^{3+}$ and $[Cu(NH_3)_4]^{2+}$

(C) $[Ag(NH_3)_2]^{+}$ and $[Cu(NH_3)_4]^{2+}$ (D) $[Ag(NH_3)_2]^{+}$ and $[Ni(NH_3)_6]^{2+}$

SA0159

27. Which of the following hydrogen halides react(s) with $AgNO_3$(aq) to give a precipitate that dissolves in $Na_2S_2O_3$(aq): **[JEE 2012]**

(A) HCl (B) HF (C) HBr (D) HI

SA0160

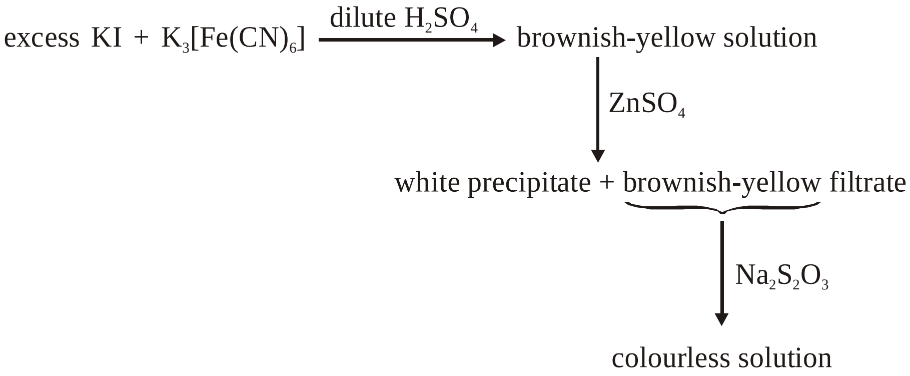
28. The reaction of white phosphorus with aqueous NaOH gives phosphine along with another phosphorus containing compound. The reaction type ; the oxidation states of phosphorus in phosphine and the other product are respectively **[JEE 2012]**

(A) redox reaction ; –3 and –5 (B) redox reaction ; +3 and +5

(C) disproportionation reaction ; –3 and +1 (D) disproportionation reaction ; –3 and +3

SA0161

29. For the given aqueous reactions, which of the statement(s) is (are) true ? **[JEE 2012]**



(A) The first reaction is a redox reaction.

(B) White precipitate is $Zn_3[Fe(CN)_6]_2$.

(C) Addition of filtrate to starch solution gives blue colour.

(D) White precipitate is soluble in NaOH solution.

SA0162

30. Upon treatment with ammonical H_2S , the metal ion that precipitates as a sulfide is -

(A) Fe(III) (B) Al(III) (C) Mg(II) (D) Zn (II) **[JEE 2013]**

SA0163

Paragraph for Question 31 and 32

An aqueous solution of a mixture of two inorganic salts, when treated with dilute HCl, gave a precipitate **(P)** and a filtrate **(Q)**. The precipitate **(P)** was found to dissolve in hot water. The filtrate **(Q)** remained unchanged, when treated with H_2S in a dilute mineral acid medium. However, it gave a precipitate **(R)** with H_2S in an ammoniacal medium. The precipitate R gave a coloured solution **(S)**, when treated with H_2O_2 in an aqueous NaOH medium. **[JEE 2013]**

31. The coloured solution **(S)** contains

(A) $Fe_2(SO_4)_3$ (B) $CuSO_4$ (C) $ZnSO_4$ (D) Na_2CrO_4

SA0164

32. The precipitate **(P)** contains

(A) Pb^{2+} (B) Hg_2^{2+} (C) Ag^{+} (D) Hg^{2+}

SA0164

33. Consider the following list of reagents : **[JEE Adv. 2014]**

Acidified $K_2Cr_2O_7$, alkaline $KMnO_4$, $CuSO_4$, H_2O_2 , Cl_2, O_3, $FeCl_3$, HNO_3 and $Na_2S_2O_3$. The total number of reagents that can oxidise aqueous iodide to iodine is

SA0165

34. Among PbS, CuS, HgS, MnS, Ag_2S, NiS, CoS, Bi_2S_3 , and SnS_2 the total number of **BLACK** coloured sulphides is **[JEE Adv. 2014]**

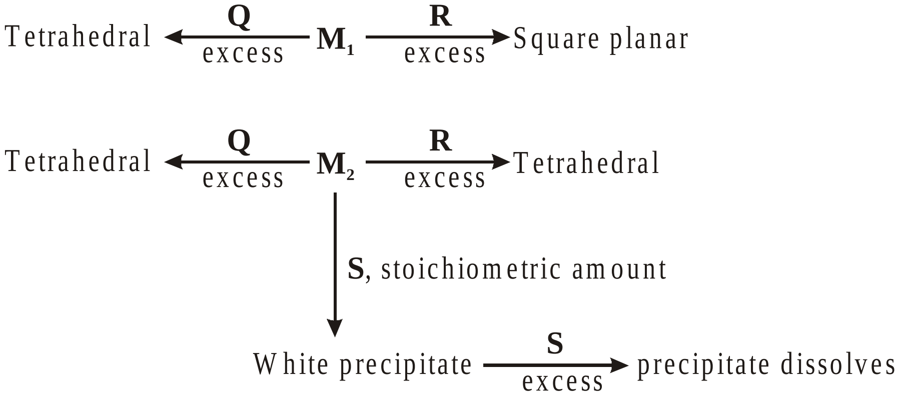
SA0166

Paragraph for Q.No. 35 and 36

An aqueous solution of metal ion $\mathbf{M_1}$ reacts separately with reagents **Q** and **R** in excess to give tetrahedral and square planar complexes, respectively. An aqueous solution of another metal ion $\mathbf{M_2}$ always forms tetrahedral complexes with these reagents. Aqueous solution of $\mathbf{M_2}$ on reaction with reagent **S** gives white precipitate which dissolves in excess of **S**. The reactions are summarized in the scheme given below.

[JEE Adv. 2014]

SCHEME:



$$\begin{tikzcd}
\text{Tetrahedral} & \mathbf{M_1} \arrow[l, "\mathbf{Q}"', "\text{excess}"] \arrow[r, "\mathbf{R}", "\text{excess}"'] & \text{Square planar} & \\
\text{Tetrahedral} & \mathbf{M_2} \arrow[l, "\mathbf{Q}"', "\text{excess}"] \arrow[r, "\mathbf{R}", "\text{excess}"'] \arrow[d, "\mathbf{S}\text{, stoichiometric amount}"] & \text{Tetrahedral} & \\
& \text{White precipitate} \arrow[r, "\mathbf{S}", "\text{excess}"'] & \text{precipitate dissolves} &
\end{tikzcd}$$

35. $\mathbf{M_1}$, **Q** and **R**, respectively are

(A) Zn^{2+}, KCN and HCl (B) Ni^{2+}, HCl and KCN

(C) Cd^{2+} , KCN and HCl (D) Co^{2+}, HCl and KCN

SA0167

36. Reagent **S** is

(A) $K_4[Fe(CN)_6]$ (B) Na_2HPO_4 (C) K_2CrO_4 (D) KOH

SA0167

37. Fe^{3+} is reduced to Fe^{2+} by using - **[JEE Adv. 2015]**

(A) H_2O_2 in presence of NaOH (B) Na_2O_2 in water

(C) H_2O_2 in presence of H_2SO_4 (D) Na_2O_2 in presence of H_2SO_4

SA0168

38. The pair(s) of ions where BOTH the ions are precipitated upon passing H_2S gas in presence of dilute HCl, is(are) **[JEE Adv. 2015]**

(A) Ba^{2+}, Zn^{2+} (B) Bi^{3+}, Fe^{3+} (C) Cu^{2+}, Pb^{2+} (D) Hg^{2+}, Bi^{3+}

SA0169

39. The reagent(s) that can selectively precipiate S^{2-} from a mixture of S^{2-} and SO_4^{2-} in aqueous soltuion is(are) : **[JEE(Adv.)-2016]**

(A) $CuCl_2$ (B) $BaCl_2$ (C) $Pb(OOCCH_3)_2$ (D) $Na_2[Fe(CN)_5NO]$

SA0170

40. In the following reaction sequence in aqueous soluiton, the species **X**, **Y** and **Z** respectively, are - **[JEE(Adv.)-2016]**

$$S_2O_3^{2-} \xrightarrow{Ag^+} \underset{\substack{\text{clear}\\ \text{solution}}}{X} \xrightarrow{Ag^+} \underset{\substack{\text{white}\\ \text{precipitate}}}{Y} \xrightarrow{\text{With time}} \underset{\substack{\text{black}\\ \text{precipitate}}}{Z}$$

(A) $[Ag(S_2O_3)_2]^{3-}$, $Ag_2S_2O_3$, Ag_2S (B) $[Ag(S_2O_3)_3]^{5-}$, Ag_2SO_3, Ag_2S

(C) $[Ag(SO_3)_2]^{3-}$, $Ag_2S_2O_3$, Ag (D) $[Ag(SO_3)_3]^{3-}$, Ag_2SO_4, Ag

SA0171

41. Which of the following combination will produce H_2 gas ? **[JEE(Adv.)-2017]**

(A) Zn metal and NaOH(aq) (B) Au metal and NaCN(aq) in the presence of air

(C) Cu metal and conc. HNO_3 (D) Fe metal and conc. HNO_3

SA0172

42. Addition of excess aqueous ammonia to a pink coloured aqueous solution of $MCl_2 . 6H_2O$ (X) and NH_4Cl gives an octahedral complex Y in the presence of air. In aqueous solution, complex Y behaves as 1 : 3 electrolyte. The reaction of X with excess HCl at room temperature results in the formation of a blue coloured complex Z. The calculated spin only magnetic moment of X and Z is 3.87 B.M., whereas it is zero for complex Y. **JEE(Adv.)-2017]**

Among the following options, which statements is(are) correct ?

(A) The hybridization of the central metal ion in Y is d^2sp^3

(B) Z is tetrahedral complex

(C) Addition of silver nitrate to Y gives only two equivalents of silver chloride

(D) When X and Z are in equilibrium at 0°C, the colour of the solution is pink

SA0173

43. The correct option(s) to distinguish nitrate salts of Mn^{2+} and Cu^{2+} taken separately is (are) :-

(A) Mn^{2+} shows the characteristic green colour in the flame test **JEE(Adv.)-2018]**

(B) Only Cu^{2+} shows the formation of precipitate by passing H_2S in acidic medium

(C) Only Mn^{2+} shows the formation of precipitate by passing H_2S in faintly basic medium

(D) Cu^{2+}/Cu has higher reduction potential than Mn^{2+}/Mn (measured under similar conditions)

SA0174

44. The green colour produced in the borax bead test of a chromium(III) salt is due to-

[JEE(Adv.)-2019]

(1) $Cr(BO_2)_3$ (2) CrB (3) $Cr_2(B_4O_7)_3$ (4) Cr_2O_3

SA0175

ANSWER KEY

EXERCISE # I

1. (A) 2. (C) 3. (C) 4. (D) 5. (D) 6. (A,B)
7. (A) → R,S; (B) → Q; (C) → P,Q,S,T; (D) → P,Q,S,T 8. (1)
9. (D) 10. (D) 11. (C) 12. (D) 13. (B) 14. (C) 15. (B)
16. (A) 17. (C) 18. (B) 19. (B) 20. (D) 21. (C) 22. (A)
23. (B) 24. (B) 25. (D) 26. (B) 27. (C) 28. (A,B) 29. (A)
30. (C) 31. (2) 32. (4) 33. (2) 34. (D) 35. (B) 36. (D)
37. (C) 38. (C) 39. (C,D) 40. (B) 41. (B) 42. (D) 43. (D)

EXERCISE # II

1. (B) 2. (C) 3. (C) 4. (A) 5. (B) 6. (B) 7. (B)
8. (B) 9. (B) 10. (D) 11. (B) 12. (B) 13. (A,B,C,D) 14. (C)
15. (C) 16. (C) 17. (B) 18. (B) 19. (B) 20. (D) 21. (C)
22. (B) 23. (B) 24. (D) 25. (C) 26. (B) 27. (C) 28. (C)
29. (C) 30. (B) 31. (A) 32. (B) 33. (D) 34. (D) 35. (C)
36. (B) 37. (D) 38. (C) 39. (D) 40. (A) 41. (C) 42. (A,C,D)
43. (C) 44. (A) 45. (A,B,C) 46. (B,C,D) 47. (A,C,D) 48. (B,D) 49. (D)
50. (A) 51. (D) 52. (D) 53. (A,C) 54. (D) 55. (B) 56. (C)
57. (B) 58. (A,B) 59. (D) 60. (B) 61. (B) 62. (B) 63. (A)
64. (A) 65. (C) 66. (B) 67. (C) 68. (D)
69. (A) → Q , R; (B) → Q ; (C) → P ; (D) → S , T 70. (A) → P ; (B) → S ; (C) → R ; (D) → Q, R
71. (B) 72. (D) 73. (C) 74. (3) 75. (2)

EXERCISE # JEE MAINS

1. (4) 2. (4) 3. (4) 4. (2) 5. (2) 6. (2) 7. (1)
8. (2) 9. (2) 10. (3) 11. (4) 12. (4) 13. (3) 14. (4)
15. (3) 16. (1) 17. (4) 18. (2) 19. (3) 20. (4) 21. (1)
22. (4) 23. (3) 24. (1) 25. (4) 26. (3) 27. (4) 28. (2)
29. (3) 30. (2) 31. (4) 32. (2) 33. (4)

EXERCISE # J-ADVANCED

1. (A, B, C) 2. (A, B, D) 3. (D) 4. (C) 5. (A) 6. (B) 7. (A)
8. (B) 9. (A) 10. (B) 11. (D) 12. (A) 13. (A)
14. (A) → P , S ; (B) → R ; (C) → P , Q ; (D) → P 15. (B) 16. (A),(B) 17. (D)
18. (C) 19. (B) 20. (A) → P,S; (B) → Q,S; (C) → R,T; (D) → Q, T 21. (A)
22. (A,C,D) 23. (B,C,D) 24. (B) 25. (A) 26. (C) 27. (A,C,D) 28. (C)
29. (A,C,D) 30. (D) 31. (D) 32. (A) 33. (7) 34. (6) / (7) 35. (B)
36. (D) 37. (A, B) 38. (C,D) 39. (A OR A, C) 40. (A) 41. (A)
42. (A,B,D) 43. (B,D) 44. (1)