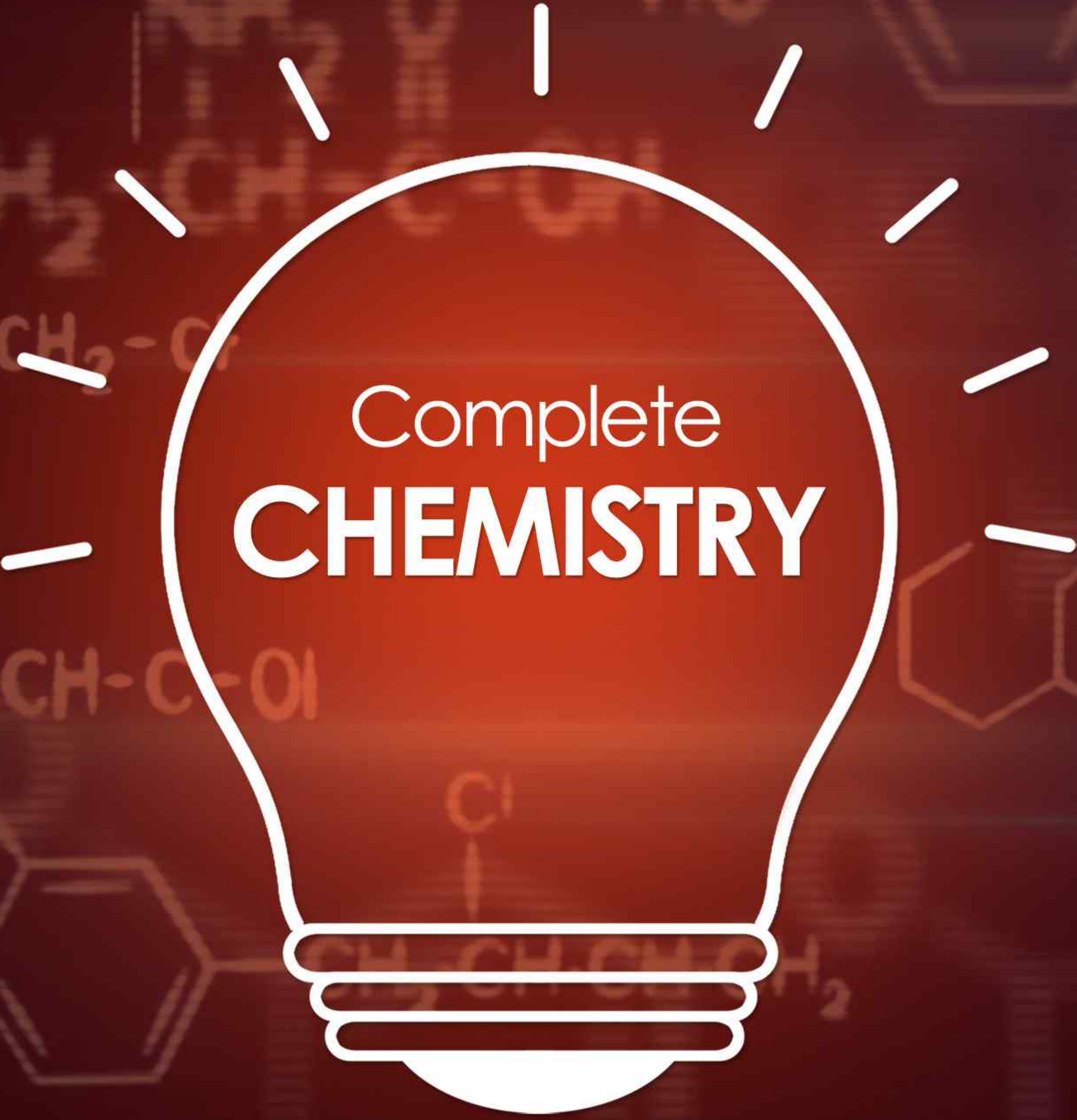
misostudy.com
smart learning



Complete
CHEMISTRY

IIT-JEE ·NEET ·CBSE eBOOKS

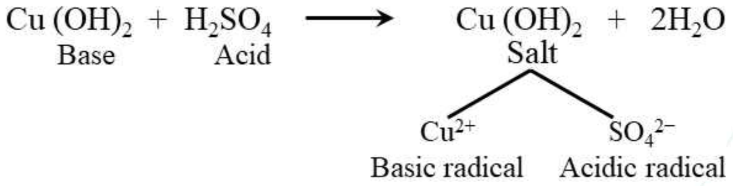
CLASS 11&12th

CLASS 12th

Salt Analysis

01. Introduction

The positively charged part of a salt (cation) which has been derived from a base is termed basic radical and the negatively charged part of the salt (anion) which has been derived from an acid is termed acidic radical.



$$\underset{\text{Base}}{Cu(OH)_2} + \underset{\text{Acid}}{H_2SO_4} \longrightarrow \underset{\text{Salt}}{Cu(OH)_2} + 2H_2O$$

$$\underset{\text{Basic radical}}{Cu^{2+}} \qquad \underset{\text{Acidic radical}}{SO_4^{2-}}$$

A mixture may have two or more salts; thus qualitative inorganic analysis consists in identifying the presence of various cations and anions in it.

02. Identification of Acidic Radicals

Group I : This group consists of radicals which are detected by dilute H_2SO_4 or dilute HCl. These are (i) carbonate, (ii) sulphite, (iii) sulphide, (iv) nitrite and (v) acetate

Group II : This group consists or radicals which are detected by concentrated H_2SO_4. These are (i) chloride, (ii) bromide, (iii) iodide, (iv) nitrate and (v) oxalate

Group III : This radicals which do not give any characteristic gas with dilute and concentrated H_2SO_4. These are (i) sulphate, (ii) phosphate, (iii) borate and (iv) fluoride.

Group I

Take 0.2 g of the substance in a test tube and add 2mL or dilute HCl or dilute H_2SO_4. Observe the reaction in cold, warm gently the contents and infer as follows :

S.NO.	Observation	Inference	Confirmatory tests
(i)	Brisk effervescence in cold with evolution of colourless and odourless gas.	CO_3^{2-} (Carbonate)	Pass the gas in a test tube containing small quantity of lime water. It turns milky
(ii)	A colourless gas with suffocating odour having smell of burning sulphur.	SO_3^{2-} (Sulphite)	Moisten a piece of filter paper with acidified potassium dichromate and put it on the mouth of the test tube. It turns green.
(iii)	A colourless gas with smell of rotten eggs.	S^{2-} (Sulphide)	Moisten a piece of filter paper with lead acetate solution and place it on the mouth of the test tube. It turns black.
(iv)	A light brown gas.	NO_2^- (Nitrite)	Pass the evolved gas through ferrous sulphate solution. It turns brown. Mix the given salt or mixture with a little or KI and add dilute H_2SO_4. Evolution of violet vapours. Place the piece of filter paper on the mouth of test tube moistened with solutions of starch, potassium iodide and acetic acid. It turns blue.
(v)	Colourless vapours with smell of vinegar.	CH_3COO^- (Acetate)	To the aqueous solution of substance add neutral $FeCl_3$ solution → blood red colour. Rub the moistened salt or mixture with dry oxalic acid → smell or vinegar.

Group II

Take 0.2 g of the substance and add 2mL of concentrated H_2SO_4. warm gently, Observe the changes and draw inference as follows :

Note : With concentrated H_2SO_4, carbonates, sulphites, sulphides, nitrites and acetates also behave in the same way as with dilute H_2SO_4.

S.NO.	Observation	Inference	Confirmatory tests
(i)	Colourless gas with pungent smell which fumes in air. [Add a pinch of MnO_2 in the solution → pale green gas is evolved.]	Cl^- (Chloride)	• Bring a glass rod dipped in NH_4OH on the mouth of test tube; white fumes are formed. • Bring a glass rod dipped in silver nitrate solution on the mouth of the test tube; white curdy ppt. is formed on the rod. • **Chromyl chloride test :** To the substance in a dry test tube add three times its weight of powdered $K_2Cr_2O_7$ and conc. H_2SO_4. Heat the contents. Red vapours are evolved. Pass the vapours in a test tube containing NaOH solution. Now add acetic acid and lead acetate solution. A yellow precipitate appears.
(ii)	Raddish brown fumes which intensify on addition of MnO_2. Vapour passed in water, make it yellow.	Br^- (Bromide)	• Take aqueous extract of the substance (or extract with dilute HNO_3) and add silver nitrate solution. A light yellow precipitate appears. • To a small amount of the substance add dilute H_2SO_4. Warm and cool. Add 1 mL chloroform or carbon tetrachloride and then chlorine water with constant shaking. The chloroform layer becomes orange-brown.
(iii)	Violet pungent fumes evolved which may condense as black specks on the cooler parts of the test tube. [The violet fumes intensify on addition of MnO_2.]	I^- (Iodide)	• Place a piece of filter paper moistened with starch solution on the mouth of the test tube. The paper turns blue. • Take aqueous extract of the substance (or extract with dilute HNO_3) and add $AgNO_3$ solution. Yellow precipitate is formed which is insoluble in NH_4OH solution. • To the small amount of the substance add dil. H_2SO_4. and 1 mL of either chloroform or carbon tetrachloride and then chlorine water, shake. Chloroform layer attains violet colouration.
(iv)	Light brown vapours having pungent smell. Intensify on adding copper turnings.	NO_3^- (Nitrate)	• Take and aqueous extract of the substance in a test tube and add freshly prepared $FeSO_4$ solution. Add conc. H_2SO_4. by the side of the test tube without disturbing the solution-a brown ring is formed at the junction of two liquids.
(v)	Colourless, odourless gas which burns with blue flame at the mouth of the test tube and turns lime water milky.	$C_2O_4^{2-}$ (Oxalate)	• Take the substance, add dil. H_2SO_4 and heat till there are no more effervescences. Now add MnO_2 (solid)-brisk effervescences.

Group III

Some anions are identified by their characteristic chemical reactions. These radicals are sulphate, borate, phosphate and fluoride.

(i) **Sulphate :** Dissolve a little amount of the substance (salt or mixture) and add barium chloride solution. A white precipitate insoluble in conc. HNO_3 is formed.

Reactions with explanations :

White precipitate of barium sulphate is obtained when soluble sulphate is treated with barium chloride solution.

$$Na_2SO_4 + BaCl_2 \rightarrow 2NaCl + \underset{\text{White ppt.}}{BaSO_4}$$

The white precipitate is insoluble in conc. HNO_3. Certain chlorides, *e.g.*, NaCl and $BaCl_2$ when present in large quantities, may form a white precipitate which dissolves on dilution with water.

Silver and lead, if present, may be precipitated as silver chloride and lead chloride by the addition of barium chloride. To avoid it, barium nitrate may be used in place of barium chloride.

(ii) **Borate :** To a small quantity of the substance (salt or mixture), add a few mL of ethyl alcohol and conc. H_2SO_4. Stir the contents with a glass rod. Heat the test tube and bring the mouth of the test tube near the flame. The formation of green edged flame indicates the presence of borate.

Reactions with explanations :

When borate is heated with ethyl alcohol and H_2SO_4, ethyl borate vapours come out which burn with green edged flame.

$$2Na_3BO_3 + 2H_2SO_4 \rightarrow 2Na_2SO_4 + 2H_3BO_3$$

$$H_3BO_3 + 3C_2H_5OH \rightarrow \underset{\text{Ethyl borate}}{(C_2H_5)_3BO_3} + 2H_2O$$

In place of ethyl alcohol, methyl alcohol can also be used. This test should be performed in a test tube and not in a porcelain basin because copper or barium salts, if present, will come in contact with the flame which also give green flame.

(iii) **Phosphate :** Take about 0.2 g of the substance in a test tube and add 2 mL conc. HNO_3. Heat and add 2 mL ammonium molybdate solution. Again heat. A canary yellow precipitate indicates the presence of phosphate.

Reactions with explanations :

The canary yellow precipitate is due to the formation of ammonium phosphomolybdate.

$$Ca_3(PO_4)_2 + 2HNO_3 \rightarrow 3Ca(NO_3)_2 + 2H_3PO_4$$

$$H_3PO_3 + 12(NH_4)_2MoO_4 + 21HNO_3 \rightarrow \underset{\text{(Canary yellow ppt.)}}{(NH_4)_3 \cdot PO_4 \cdot 12MoO_3} + 21NH_4NO_3 + 12H_2O$$

Arsenic under similar conditions also yields a yellow precipitate of $(NH_4)_3 \cdot AsO_4 \cdot 12MoO_3$ (ammonium arsenomolybdate). So, in presence of As, phosphate is tested in the filtrate of second group.

(a) The precipitate of ammonium phosphomolybdate dissolves in excess of phosphate. Thus, the reagent (ammonium molybdate) should always be added in excess.

(b) HCl interferes in this test. Hence, if the test of phosphate is to be performed with the solution containing HCl, the solution should be boiled to remove HCl.