Qualitative Inorganic Analysis

Qualitative inorganic analysis is a vital aspect of chemistry, used in fields like medicine, criminology, and industry. It involves identifying radicals in inorganic salts. Inorganic salts result from acid-base reactions, with cations (basic radicals) and anions (acidic radicals). For example, in sodium chloride, Na^+ is the cation, and Cl^- is the anion.

Before testing an inorganic substance, note its color, state, and solubility in water. Color and solubility provide initial clues about cations and anions.

The analysis includes wet and confirmatory tests. Preliminary dry tests are quick and involve observing the salt's appearance, smell, and solubility. They include heating, flame tests, borax bead tests, and more.

Solubility in water and solution pH reveal ion information. Acidic or basic solutions indicate hydrolysis. Gases produced during tests with acids like H_2SO_4 provide insights about acid radicals. These dry tests are essential before confirmatory ion tests.

Color of some inorganic salts

Color	Compounds
White or colorless	Salt of Na, K, NH, Mg, Ca, Sr, Ba, Zn and Al
Blue	Hydrated cupric salt and anhydrous cobalt salt
Pink	Hydrated salt of Co and Mn
Green	Fe(ous) salt, except $FeS,\ CuCl_2,\ 2H_2O,\ CuCo_3,\ N(ous)$ salt
Black	$FeS,\ CoS,\ NiS,\ PbS$

Solubility of Inorganic Compounds in Water

Compounds	Solubility in Water
Nitrates	All salts are soluble
Sulfates	All except $Pb,\ Fe(\mathrm{ic}),\ Ba,\ Sr,\ Ca$
Nitrites	All except Ag

Chlorides, Bromides and Iodides	All chlorides, bromides and iodides except Ag, Pb and mercurous mercury (Hg_2^{2+})
Carbonates	Na, K, NH only
Sulfate	Na, K, NH only
Hydroxide	Na, K, NH only

- The qualitative analysis of an inorganic substance is generally carried out in the following four parts:
    1. Dry test for Basic Radicals
    2. Dry test for Acidic Radicals
    3. Wet test for anions (basic radicals)
    4. Wet test for cations (acidic radicals)

Dry Test for Basic Radicals

Perform this experiment by heating a small amount of salt in a dry test tube.
Information from this test can give valuable clues, partly from observation of the residue and partly from identification of any gas evolved. Tabulate your observations along with the interferences that you can draw.

> A pinch of solid is put to dry test in a test tube.

Observation	Inference
1. (Resiedue) Change in color	
(a) Yellow when hot, white when cold	Zn^{2+}
(b) Yellow in both hot and cold conditions	Pb^{2+}
(c) Red white hot and yellow or brown when cold	Fe^{3+}
(d) Black in both hot and cold conditions	Salt of $Cu^{2+},\ Co^{2+},\ Mn$
2. Salt fuses while melting	$Na^+, K^+, CA^{2+}, Sr^{2+}, Ba^{2+}$
3. Decrepitation (the salt flies to pieces with sharp crackling sound)	$Pb(NO_3),\ NaCl$

4. Formation of sublimate (white colored along with fog)	NH_4^+
5. Evolution of gases	
(a) Colorless and odorless gas, turns lime water milky	CO_3^{2-} or $C_2O_4^{3-}$ present
(b) Evolution of ammonia, gives white fumes when a glass rod dipped in dilute HCl is brought near the mouth of the test tube.	NH_4^+

Dry Test For Acid Radicals

- Dry tests for acid radicals are performed in the following manner
    1. Action of diluted hydrochloric acid
    2. Action of concentrated sulfuric acid

Action of Diluted Hydrochloric Acid

Take a small amount of the substance and add 5 ml of diluted H_2SO_4, if no reaction takes place, warm a little.

Observation	Gas Evolved	Possible anion
1. Colorless and odorless gas is evolved with fizz, turns lime water milky.	CO_2	Carbonate (CO_3^{2-}) and Bicarbonate (HCO_3^-)
2. Colorless gas with odor of rotten eggs turns lead acetate paper black.	H_2S	Sulfide (S^{2-})
3. Colorless gas with pungent smell, like burning sulfur which turns acidified potassium dichromate solution green.	SO_2	Sulfite (SO_3^{2-})
3. Reddish-brown gas is evolved turns acidified startch-iodide paper blue (starch iodide paper is prepared by moistening a piece of filter paper with a Kl solution)	Cl_2	Nitrite (NO_2^-)

4. Colourless vapours with smell of vinegar. Vapours turn blue litmus red.	CH_3COOH vapours	Acetate, (CH_3COO^-)

Action of Concentrated Sulfuric Acid

Take a small amount of substance in a dry test tube. Add 4-5 ml of concentrated H_2SO_4, and gently heat the solution.

Observation	Gas evolved	Possible anions
1. A colourless gas with pungent smell, which gives dense white fumes when a rod dipped in NH_4OH (ammonium hydroxide) is brought near the mouth of the test tube	HCl	Chloride, Cl^-
2. 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 vapors	Bromide, Br^-
3. 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 vapors	Iodide, I^-
4. 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^-
5. 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 $(C_2O_4^{2-})$

Take a small amount of substance in a dry test tube. Add a little MnO_2 and 2 ml concentrated $H2SO4$. Heat gently.

Observation	Gas evolved	Possible anions
1. Greenish-yelow gas turns moist starch-iodide paper blue.	Cl_2	Chloride (Cl^-)

2. Brown-red vapors turn strach-iodide paper blue.	Br_2	Bromide (Br^-)
3. Violet vapors turn starch paper blue.	I_2	Iodide (I^-)

Take a small amount of the substance in a dry test tube. Add 3-4 ml concentrated H_2SO_4 and a little Cu turning. Heat gently.

Observation	Gas evolved	Possible Anion
Reddish-brown fumes and the solution in the test tube appears blue.	NO_2	Nitrate (NO_3^-)

Wet Test for Acid Radical

> If salt not soluble in H_2O, then prepare Na_2CO_3 extract. Otherwise water extract.

Experiment	Observation	Inference
1. take about 5 ml of NA_2CO_3 extract in a test tube. Acidify with dilute HCl. Boil to expel CO. Add $BaCl_2$ solution.	White ppt insoluble in dilute HCl or dilute HNO_3	Sulfate $(SO_4)^{2-}$
2. Take 5 ml of Na_2CO_3 (sodium carbonate) extract in a test tube. Accidiryfwith dilute HCl. Add $BaCl_2$ solution followed by bromine water. Heat the content.	White ppt	Slufite (SO_3^{2-})
3. Take 5 ml of sodium carbonate extract in a test tube. Acidify with dilute H_2SO_4. Add about 5 ml of freshly prepared $FeSO_4$ solution. Slowly pour 2-3 ml of concentrated H_2SO_4 down the side of the tube. Hold the tube at an angle while doing this. The heavy sulfuric acid sinks to the bottom.	A brown ring at the junction of the two liquids.	Nitrate (NO_3^-)

4. Take 5 ml of Na_2CO_3 extract in a test tube. Acidify with dilute HNO_3. Boil off CO_2. Add $AgNO_3$ solution.	**1.** White ppt readily soluble in NH_4OH	Chloride (CL^-)
	2. Pale yellow ppt soluble in concentrated NH_4OH solution.	Bromide Br^-
	3. Yellow ppt insoluble in concentrated NH_4OH	Iodide (I^-)
5. Take 5 ml of Na_2CO_3 extract. Acidify with CH_3COOH (acetic acid) and then add $Pb(C_2H_3O_2)_2$ (lead acetate) solution.	Black ppt soluble in hot dilute HNO_3	Sulfide (S^2-)
6. Take 5 ml of Na_2CO_3 extract, add a few drops of $Na_2[Fe(CN)_5(NO)]$ (sodium nitroprusside) solution.	Violet or pink coloration	Sulfide (S^2-)
7. Take 5 ml of Na_2CO_3 extract in a test tube. Acidify with dilute H_2SO_4. Then add KI (Potassium iodide) solution.	Reddish-brown coloration turns starch solution blue.	Nitrate (NO_2^-)
8. Take 5 ml of Na_2CO_3 extract in a test tube. Add 2-3 ml of concentrated HNO_3 and 10 ml of $(NH_4)_6Mo_7O_{24}$ (ammonium molybdate) solution. Warm moderately $(40^\circ C)$	Straw-colored ppt (light-yellow)	Phosphate (PO_4^{3-})
9. Take 2-3 ml of Na_2CO_3 extract in a test tube. Acidify with dilute CH_3COOH (acetic acid). Add $CaCl_2$ solution.	White ppt insoluble in dilute HCl	Oxalate $(C_2O_4^{2-})$
10. Dissolve salt in water: To the aqueous solution, add freshly prepared $FeCl_2$ solution.	Blood red coloration	Acetate (CH_3COO)

Wet Test for Basic Radical

1. Distilled water (hot/cold)
2. Dilute HCl (hot/cold)
3. Concentrated HCl (hot/cold)

Solubility of inorganic salt in the above solvents may often help to identify some basic radicals.

Solvent	Salt
1. Cold water	(a) All NH_4, Na and K salts.
	(b) All nitrates, nitrites and acetates
	(c) Most of the sulfates except those of $Pb,\ Ba,\ Ca,\ Sr$
	(d) All chlorides exceps that of lead
2. Hot water	Lead chloride, lead nitrate
	All carbonates which don't dissolve in water, i.e., carbonates of $Ca,\ Ba,\ Sr,\ Mg,\ Fe,\ etc$ but not of PbS

The usual procedures for analyzing a salt involves the systematic separation of basic radicals into groups. The table given below gives briefly the classification of basic radicals into groups of reagents and the form in which they are precipitated.

Group	Group Reagent	Basic Reagent	Form in which basic radicals are precipitated
1.	Dilute HCl	$Pb^{2+},\ Hg^{2+}(\text{ous}),\ Ag^{+}$	Chlorides
2.	H_2S in presence of dilute HCl	$Pb^{2+},\ Cu^{2+},\ As^{3+},\ Hg^{2+},\ Cd^{2+},\ Bi^{3+},\ Sb^{3+},\ Sn^{2+}$	Sulfides
3.	NH_4OH in presence of NH_4Cl	$Fe^{3+},\ Al^{3+},\ Cr^{3+}$	Hydroxides

4.	H_2S in presence of NH_4OH	$Ni^{2+},\ Mn^{2+},\ Zn^{2+},\ Co^{2+}$	Sulfides
5.	$(NH_4)_2CO_3$ in the presence of NH_4OH	$Ca^{2+},\ SrA^{2+},\ Be^{2+}$	Carbonates
6.	---	$Mg^{2+},\ Na,\ K$	---

Division of the Basic Radicals (cations) in Different Groups (group separation)

Experiments	Observation	Inference
1. Add dilute HCl to the salt solution.	White ppt	Group I present
2. Add dilute HCl to the salt solution and then pass H_2S gas	Black ppt	Group 2 present
3. Boil salt solution with salt + HCl and add NH_4Cl and NH_4OH solution in excess		Group 3 present
	(a) Reddish-brown ppt	Fe salt
	(b) White gelatinous ppt	Al salt
4. Add NH_3Cl and NH_4OH to salt solution and pass H_2S gas		Group 4 present
	(a) Black ppt	Co salt
	(b) Pink ppt	Mn salt
	(c) White ppt	Zn salt
5. Add NH_4OH and $(NH_4)_2CO_3$ to the salt solution	White ppt	Group 5 present maybe $Ca,\ Ba,\ Sr$ salt
6. No particular group reagent	---	Group 6 Mg salt