

A' LEVEL BASIC QUALITATIVE ANALYSIS EXPOSED

INORGANIC COMPOUNDS

Qualitative analysis is the determination of **elements or ions** present in a given substance by carrying out specific tests and making keen observations of the changes that take place.

In this case the commonly observed changes include the following:

- Color changes
- Precipitate formed
- Gases evolved
- Sound
- Smell etc.

AN ION

An ion is an atom or a group of atoms that have charges. The charges may either be positive or negative

TYPE OF IONS

There are **two types** of ions

- i. **Cations:** These are positively charged ions. The common cations identified in A' level chemistry include;
 $Ca^{2+}, Mg^{2+}, Cu^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, NH_4^+, Fe^{2+}, Fe^{3+}, Ba^{2+}, Ni^{2+}, Ag^+, Co^{2+}, Mn^{2+}, Cr^{3+}, Sn^{2+}$
- ii. **Anions:** These are negatively charged ions. The common anions identified in A' level chemistry include the following; $Cl^-, SO_4^{2-}, CO_3^{2-}, NO_3^-, HCO_3^-, SO_3^{2-}, Br^-, I^-, C_2O_4^{2-}, CrO_4^{2-}, S^{2-}, CH_3COO^-, S_2O_3^{2-}$.

IDENTIFICATION OF UNKNOWNNS

1. APPEARANCE

Note the color, smell if any and the physical state of the given salt sample or substance

Appearance of the solid sample(color) (observation)	Deduction
i. Black	Oxide of Copper, Cu^{2+} probably present or Sulphides of Cu^{2+} , Ni^{2+} , Ag^+ , Co^{2+} , Pb^{2+} or Fe^{2+}
ii. Blue	Hydrated salt of copper, Cu^{2+} , Ni^{2+} , Cr^{3+} or Fe^{2+} probably present or Co^{2+} (an hydrous)
iii. Green	Hydrated salts of Copper (II), Nickel (II), Chromium (III) or Iron (II), Cu^{2+} , Ni^{2+} , Cr^{3+} or Fe^{2+} probably present.
iv. Yellow/brown	Lead(II)oxide(in case of solid) or Iron(III) salt probably present
v. White solid	Salts of Ca^{2+} , Mg^{2+} , Pb^{2+} , Al^{3+} , Zn^{2+} , NH_4^+ , Ba^{2+} , probably present
vi. Orange-red color	Cr^{6+} (from a dichromate)
vii. Purple	Mn^{7+} (from a permanganate)
viii. Pink	Hydrated salts of Manganese, Mn^{2+} present
ix. Yellow color	Chromate or ferric salt
x. Pink or red	Hydrated salt of Mn^{2+} or Co^{2+}

2. SMELL/ODOUR

Very few inorganic salts have recognizable Odours at room temperature. When a small amount of the unknown is gently heated; the following characteristic smells may be detected.

(Smell) Observation	Deduction
Smell of ammonia (pungent, chocking smell "smell of urine")	Ammonium salt, NH_4^+ present
Smell of hydrogen sulphide (pungent, rotten egg smell) NB. H_2S is poisonous, smell with care	S^{2-} present
Smell of sulphur dioxide (poisonous, sharp chocking smell of burning sulphur), smell with care	Normal and acid sulphates, thiosulphate or sulphites

Smell of chlorine (pungent, poisonous and bleaches, smell with care)	Bleaching powder (CaOCl_2)
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NB:

- If the substance is a **crystalline solid** then; Cl^- , SO_4^{2-} , NO_3^- , $\text{C}_2\text{O}_4^{2-}$, $\text{S}_2\text{O}_3^{2-}$ is probably present
- If the substance is in **powder** form then **carbonates or oxides** are more likely
- If the substance is **deliquescent** then **chloride ion** is probably present

3. ACTION OF HEAT ON A SUBSTANCE

Heat a little of the substance in a dry test tube until no further change occurs, Note what happens and test for the gas evolved.

a) GASES EVOLVED DURING HEATING

Observation	Deduction
i. Colourless liquid condenses at the cooler parts of the test-tube which is neutral to litmus paper and turns white anhydrous copper(II) sulphate blue	Water of crystallization(hydrated salt) present or may be HCO_3^- , OH^- or HSO_4^- present
ii. Brown fumes of a gas which turn damp blue litmus paper red and darkens FeSO_4 solution, does not bleach litmus are evolved	NO_2 gas, NO_3^- probably present
iii. Colourless gas with a pungent choking smell which turns damp red litmus paper blue is evolved and forms white fumes with HCl gas. White sublimate formed on the middle part of the test tube	Ammonia gas produced, NH_4^+ present
iv. Colourless, odourless gas, neutral to litmus which relights a glowing splint is given off	Oxygen gas produced, O^{2-} , ClO_3^- , NO_3^- , peroxides (e.g. H_2O_2), higher oxides probably present
v. Colourless gas which turns damp blue litmus paper red(pink) and lime water milky	CO_2 gas produced, CO_3^{2-} or HCO_3^- or $\text{C}_2\text{O}_4^{2-}$ probably present

vi.	Colourless gas with pungent chocking smell which forms white fumes in air and turns damp blue litmus red and $AgNO_3$ solution milky. Forms dense white fumes with Conc. NH_3 solution	HCl produced, Cl^- present
vii.	White (smoky) fumes, turns damp blue litmus red and $BaCl_2$ or $Ba(NO_3)_2$ solution milky	SO_3 produced, HSO_4^- or SO_4^{2-} present
viii.	Colourless gas with sharp chocking smell (like burning sulphur), turns blue litmus red, turns orange $Cr_2O_7^{2-}$ solution to green and purple MnO_4^- solution to colourless	SO_2 gas produced
ix.	Crystalline solid melts on strong heating to give a cream solid on cooling	SO_3^{2-} present SO_3^{2-} decomposed to sulphur and SO_2 gas
x.	Crystalline solid formed white residue which turns yellow on strong heating, SO_2 gas may also be produced.	$S_2O_3^{2-}$ present $S_2O_3^{2-}$ decomposed to sulphur (yellow) which melts to thick black liquid.
xi.	Yellow residue formed, turned brown and gave a black viscous liquid	$S_2O_3^{2-}$ present $S_2O_3^{2-}$ decomposed to sulphur (yellow) which melts to thick black liquid.

b) NATURE OF RESIDUE FORMED DURING/ AFTER HEATING

Observation	Deduction
i. Residue is yellow when hot and white when cold	ZnO formed, Zn^{2+} present
ii. Residue is brown(Red) when hot and yellow when cold	PbO formed, Pb^{2+} present
iii. The solid turns from blue/ green to black	CuO is formed, Cu^{2+} present
iv. The solid turns from blue to white. Droplets of a colorless liquid are formed at the cooler parts of the test-tube	Anhydrous Copper (II) sulphate is formed. Water of crystallization(hydrated salt) present

v.	Green solid turns black	CuO , FeO , or NiO formed, Cu^{2+} , Fe^{2+} or Ni^{2+} suspected present
vi.	Pink to blue, then to black on strong heating	Co^{2+} present CoO formed from Hydrated Co^{2+}
vii.	Yellow-brown solid to red-brown solid	Fe^{3+} present Fe_2O_3 formed
viii.	Crystalline orange to green	Cr^{6+} (from $Cr_2O_7^{2-}$) Cr_2O_3 formed
ix.	Red brown solid forms red liquid on heating, yellow glassy residue remained. A colourless gas that relights a glowing splint is produced	Pb^{4+} , (PbO_2) reduced to Pb^{2+} (in PbO) O_2 gas produced
x.	Red-orange solid initially darkens on heating, then melts to a red liquid which cools to form a yellow residue. A colourless gas that relights a glowing splint is produced	Pb_2O_4 (PbO or PbO_2) decomposed to PbO O_2 gas produced
xi.	No observable change	The solid is thermally stable, O^{2-} or SO_4^{2-} probably present

4. IDENTIFICATION OF GAS

This is also very important in qualitative analysis because it gives clue to the ions present in the given sample

Gas	Color and smell	Test	Observation
Carbon dioxide (acidic)	Colourless and odourless	Bubble gas into lime water	Lime-water turns milky
Oxygen	Colourless and odourless	Lowering a glowing splint in a tube	Splint rekindles (relights)
Ammonia (alkaline)	Colourless and chocking smell	- Expose to damp red litmus paper - Expose to hydrogen chloride fumes	- Litmus paper turns blue - Dense white fumes are formed
Nitrogen dioxide (acidic)	Brown fumes and irritating smell	Expose to damp blue litmus paper	Litmus paper turns red

Hydrogen chloride (acidic)	Colourless and irritating smell	Expose to ammonia fumes	Dense white fumes
Sulphur dioxide (acidic)	Colourless	- Bubble it through acidified $K_2Cr_2O_7$ - Or acidified $KMnO_4$	$K_2Cr_2O_7$ turns from orange to green $KMnO_4$ turns from purple to colourless

NB:

- All acidic gases turn damp blue litmus paper to red while alkaline gases e.g. ammonia gas turns red litmus blue.

5. SOLUBILITY OF A SUBSTANCE

Here take note of whether the substance completely or partly dissolves in a given solvent (e.g. water). Also take note of the colour of the resultant solution formed.

Observation	Deduction
i. White solid dissolves forming a colourless solution	$Ca^{2+}, Mg^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, NH_4^+, Ba^{2+}, Mn^{2+}$ (very pale pink) probably present
ii. Brown/yellow solid dissolves forming a brown or yellow solution which turns damp blue litmus red.	Fe^{3+} present
iii. Green solid dissolves forming a green solution.	Hydrated salt of $Fe^{2+}, Cu^{2+}, Ni^{2+}$ or Cr^{3+} probably present
iv. Blue solid dissolves giving a blue solution.	Hydrated salt of $Cu^{2+}, Ni^{2+}, Cr^{3+}$ or Fe^{2+} probably present.
v. Yellow solid dissolves giving a yellow solution	Fe^{3+} or Cr^{6+} (from CrO_4^{2-}) suspected present
vi. An orange solid dissolves giving an orange solution	Cr^{6+} (from $Cr_2O_7^{2-}$) suspected present
vii. Purple solid dissolves giving a purple solution	Mn^{7+} (from a permanganate) or Cr^{3+} (from conc. Solutions)
viii. Pink or red solid dissolves giving pink or	Hydrated Co^{2+} or Mn^{2+} (very pale pink usually invisible in solution)

	red solution	
ix.	Pale green solid dissolves forming a pale green solution.	Hydrated salt of Fe^{2+} , Cu^{2+} or Cr^{3+} probably present

N.B

- All **nitrates** are **soluble** in water
- All K^+ , NH_4^+ , Na^+ **salts** are soluble in water
- All **carbonates** are **insoluble** in water except carbonates of potassium, sodium and ammonium.
- All **sulphates** are soluble except $CaSO_4$ and Ag_2SO_4 which are sparingly soluble. $BaSO_4$ and $PbSO_4$ are insoluble in water
- All **sulphites** are **soluble** except sulphites of Ca^{2+} , Ba^{2+} and Pb^{2+} .
- All **chlorides** are **soluble** in water except $PbCl_2$ which is sparingly soluble and $AgCl$ which is insoluble in water
- All **chromates** are **soluble** except; $CaCrO_4$ is moderately soluble and $PbCrO_4$, $BaCrO_4$ and Ag_2CrO_4 are insoluble
- All oxalates and phosphates are insoluble except oxalates and phosphates of Mg^{2+} , K^+ , NH_4^+ and Na^+ .
- All **hydroxides** are **insoluble** except those of **sodium, potassium and ammonium**. **Magnesium** and **calcium** hydroxides are sparingly soluble in water.

6. DETECTION OF CATIONS

Cations are detected by use of Sodium hydroxide and Ammonia solution. In this case take note of whether the precipitate is formed or not and record the colour of the precipitate formed. Take note of whether also the precipitate dissolves in excess or not

a) Addition of sodium hydroxide solution

Ion	Test	Observation and equation
NH_4^+	To a solution of NH_4^+ ions in a test-tube add NaOH drop wise until in excess	No observable change. A colorless gas with a pungent choking smell is given off on warming. The gas turns damp red litmus paper blue and forms white fumes with Conc. HCl Ionic equation: $NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(g) + H_2O(l)$

Sn^{2+}	To a solution of Sn^{2+} ions in a test-tube add NaOH drop wise until in excess	White precipitate soluble in excess to form a colourless solution Ionic equation: $\text{Sn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Sn}(\text{OH})_2(\text{s})$ White ppt $\text{Sn}(\text{OH})_2(\text{s}) + 4\text{OH}^{-}(\text{aq}) \rightarrow \text{Sn}(\text{OH})_6^{4-}(\text{aq})$ Stannate (II) ion (colourless)
Zn^{2+}	To a solution of Zn^{2+} ions in a test-tube add NaOH drop wise until in excess	White precipitate soluble in excess to form a colorless solution Ionic equation: $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ White ppt $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Zn}(\text{OH})_4^{2-}(\text{aq})$ Zincate ion (colourless)
Mn^{2+}	To a solution of Mn^{2+} ions in a test-tube add NaOH drop wise until in excess	White precipitate insoluble in excess, darkens and turns brown on standing due to formation of MnO_2 or Mn_2O_3 Ionic equation: $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ White ppt <u>On standing:</u> $2\text{Mn}(\text{OH})_2(\text{s}) + \text{O}_2(\text{g}) \rightarrow 2\text{MnO}_2 \cdot \text{H}_2\text{O}(\text{s})$ (brown ppt)

Al^{3+}	To a solution of Al^{3+} ions in a test-tube add NaOH drop wise until in excess	White precipitate soluble in excess to form a colourless solution. Ionic equation: $Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Al(OH)_3(s)$ White ppt $Al(OH)_3(s) + OH^{-}(aq) \rightarrow Al(OH)_4^{-}(aq)$ (Aluminate ion) (colourless)
Pb^{2+}	To a solution of Pb^{2+} ions in a test-tube add NaOH drop wise until in excess	White precipitate soluble in excess to form a colourless solution Ionic equation: $Pb^{2+}(aq) + 2OH^{-}(aq) \rightarrow Pb(OH)_2(s)$ White ppt $Pb(OH)_2(s) + 2OH^{-}(aq) \rightarrow Pb(OH)_4^{2-}(aq)$ Plumbate ion (colourless)
Mg^{2+} Ca^{2+}	To a solution of Mg^{2+} or Ca^{2+} ions in a test-tube add NaOH drop wise until in excess	White precipitate insoluble in excess (conc. Solutions of Mg^{2+} or Ca^{2+}) Ionic equation: $Mg^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mg(OH)_2(s)$ White ppt $Ca^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ca(OH)_2(s)$ White ppt
Ba^{2+}	To a solution of Ba^{2+} ions in a test-tube add NaOH drop wise until in excess	No observable change
Cu^{2+}	To a solution of Cu^{2+} ions in a test-tube add NaOH drop wise until in excess	A pale blue precipitate insoluble in excess Ionic equation: $Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$ A pale blue ppt N.B The pale blue ppt turns black on heating due to formation of CuO

		$\text{Cu(OH)}_2(\text{s}) \rightarrow \text{CuO}(\text{s}) + \text{H}_2\text{O}(\text{l})$
Co^{2+}	To a solution of Co^{2+} ions in a test-tube add NaOH drop wise until in excess	A pale or light blue precipitate insoluble in excess Ionic equation: $\text{Co}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Co(OH)}_2(\text{s})$ A pale blue ppt N.B The pale blue ppt turns grey-pink (or brown) on standing due to oxidation of Co^{2+} to Co^{3+}
Fe^{2+}	To a solution of Fe^{2+} ions in a test-tube add NaOH drop wise until in excess	A dirty green precipitate insoluble in excess. The dirty green precipitate turns brown on standing due to oxidation of Fe^{2+} to Fe^{3+} Ionic equation: $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Fe(OH)}_2(\text{s})$ A dirty green ppt N.B <u>Oxidation reaction</u> $4\text{Fe(OH)}_2(\text{s}) + \text{O}_2(\text{g}) \rightarrow \text{Fe}_2\text{O}_3 \cdot 2\text{H}_2\text{O}(\text{s})$
Ni^{2+}	To a solution of Ni^{2+} ions in a test-tube add NaOH drop wise until in excess	A green precipitate insoluble in excess. Ionic equation: $\text{Ni}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Ni(OH)}_2(\text{s})$ A green ppt
Fe^{3+}	To a solution of Fe^{3+} ions in a test-tube add NaOH drop wise until in excess	A brown or yellow precipitate insoluble in excess. Ionic equation: $\text{Fe}^{3+}(\text{aq}) + 3\text{OH}^{-}(\text{aq}) \rightarrow \text{Fe(OH)}_3(\text{s})$ A brown/yellow ppt
Cr^{3+}	To a solution of Cr^{3+} ions in a test-tube add NaOH drop wise until in excess	A grey-green precipitate soluble in excess giving a dark green solution

		Ionic equation: $\text{Cr}^{3+}(\text{aq}) + 3\text{OH}^{-}(\text{aq}) \rightarrow \text{Cr}(\text{OH})_3(\text{s})$ A grey-green ppt In excess: $\text{Cr}(\text{OH})_3(\text{s}) + 3\text{OH}^{-}(\text{aq}) \rightarrow \text{Cr}(\text{OH})_6^{3-}(\text{aq})$ Chromite ion (dark green solution)
$\text{Cr}_2\text{O}_7^{2-}$	To a solution of $\text{Cr}_2\text{O}_7^{2-}$ ions in a test-tube add NaOH drop wise until in excess	The solution changes from orange to yellow $\text{Cr}_2\text{O}_7^{2-}$ present, $\text{Cr}_2\text{O}_7^{2-}$ converted to CrO_4^{2-} $\text{Cr}_2\text{O}_7^{2-}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow 2\text{CrO}_4^{2-}(\text{aq}) + \text{H}_2\text{O}(\text{l})$

SUMMARY

Add sodium hydroxide solution drop-wise until in excess

Observation	Deduction
No observable change. A colourless gas with a choking smell that turns damp red litmus paper blue is evolved on heating. The gas also forms white fumes with hydrogen chloride gas	Ammonia gas, NH_4^{+} present
White precipitate soluble in excess	$\text{Pb}^{2+}, \text{Al}^{3+}, \text{Zn}^{2+}, \text{Sn}^{2+}, \text{Sn}^{4+}$ probably present
White precipitate insoluble in excess	$\text{Ca}^{2+}, \text{Mg}^{2+}$ probably present
No observable change	Ba^{2+} probably present
White ppt insoluble in excess, darkens or turns brown on standing	Mn^{2+} present
A pale blue precipitate insoluble in excess and turns black on heating	Cu^{2+} present CuO formed on heating
A dirty green precipitate insoluble in excess and turns brown on standing	Fe^{2+} present Fe^{2+} oxidized to Fe^{3+}
A brown precipitate insoluble in excess	Fe^{3+} present
A pale blue precipitate insoluble in excess and turns grey- pink (or brown) on standing	Co^{2+} present Co^{2+} oxidized to Co^{3+}
A green precipitate insoluble in excess	Ni^{2+} present

Grey-green ppt soluble in excess giving a dark green solution	Cr^{3+} present
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b) Addition of ammonia solution

Ion	Test	Observation and equation
Zn^{2+}	To a solution of Zn^{2+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate soluble in excess to form a colourless solution Ionic equation: $Zn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Zn(OH)_2(s)$ White ppt $Zn(OH)_2(s) + 4NH_3(aq) \rightarrow Zn(NH_3)_4^{2+}(aq) + 2OH^{-}(aq)$ Tetra ammine zinc(II)ion (colourless)
Al^{3+}	To a solution of Al^{3+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess. Ionic equation: $Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Al(OH)_3(s)$ White ppt
Pb^{2+}	To a solution of Pb^{2+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess Ionic equation: $Pb^{2+}(aq) + 2OH^{-}(aq) \rightarrow Pb(OH)_2(s)$ White ppt
Mg^{2+} Ba^{2+}	To a solution of (conc.) Mg^{2+} , Ba^{2+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess Ionic equation: $Mg^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mg(OH)_2(s)$ White ppt $Ca^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ca(OH)_2(s)$ White ppt
Ca^{2+}	To a solution of (conc.) Ca^{2+} ions in a test-tube add ammonia solution drop wise until in excess	No observable change

Sn^{2+}	To a solution of Sn^{2+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess. Ionic equation: $\text{Sn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Sn}(\text{OH})_2(\text{s})$ White ppt
Mn^{2+}	To a solution of Mn^{2+} ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess, darkens and turns brown on standing due to formation of MnO_2 or Mn_2O_3 Ionic equation: $\text{Mn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Mn}(\text{OH})_2(\text{s})$ White ppt On standing: $2\text{Mn}(\text{OH})_2(\text{s}) + \text{O}_2(\text{g}) \rightarrow 2\text{MnO}_2 \cdot \text{H}_2\text{O}(\text{s})$ (brown ppt)
Cu^{2+}	To a solution of Cu^{2+} ions in a test-tube add ammonia solution drop wise until in excess	A pale blue precipitate soluble in excess to form a deep solution Ionic equation: $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ A pale blue ppt $\text{Cu}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow \text{Cu}(\text{NH}_3)_4^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq})$ Tetra ammine copper(II) ions Deep blue solution
Fe^{2+}	To a solution of Fe^{2+} ions in a test-tube add ammonia solution drop wise until in excess	A dirty green precipitate insoluble in excess. The dirty green precipitate turns brown on standing due to oxidation of Fe^{2+} to Fe^{3+} Ionic equation: $\text{Fe}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Fe}(\text{OH})_2(\text{s})$ A dirty green ppt N.B Oxidation reaction $4\text{Fe}(\text{OH})_2(\text{s}) + \text{O}_2(\text{g}) \rightarrow \text{Fe}_2\text{O}_3 \cdot 2\text{H}_2\text{O}(\text{s})$
Ni^{2+}	To a solution of Ni^{2+} ions in a test-tube add ammonia solution drop wise until in excess	A green precipitate soluble in excess forming a pale blue/green solution.

		Ionic equation: $Ni^{2+}(aq) + 2OH^{-}(aq) \rightarrow Ni(OH)_2(s)$ A green ppt In excess $Ni(OH)_2(s) + 6NH_3(aq) \rightarrow Ni(NH_3)_6^{2+}(aq) + 2OH^{-}(aq)$ (a pale green solution)
Co^{2+}	To a solution of Co^{2+} ions in a test-tube add ammonia solution drop wise until in excess	Pink-blue/ dirty blue precipitate soluble in excess ammonia/ NH_4Cl solution, forms yellow-brown or red solution on standing or addition of H_2O_2 . Ionic equation: $Co^{2+}(aq) + 2OH^{-}(aq) \rightarrow Co(OH)_2(s)$ A dirty blue ppt In excess $Co(OH)_2(s) + 6NH_3(aq) \rightarrow Co(NH_3)_6^{2+}(aq) + 2OH^{-}(aq)$
Cr^{3+}	To a solution of Cr^{3+} ions in a test-tube add ammonia solution drop wise until in excess	A grey-green precipitate insoluble in excess./ slightly soluble forming pink/violet solution Ionic equation: $Cr^{3+}(aq) + 3OH^{-}(aq) \rightarrow Cr(OH)_3(s)$ A grey-green ppt
Fe^{3+}	To a solution of Fe^{3+} ions in a test-tube add ammonia solution drop wise until in excess	A brown or yellow precipitate insoluble in excess. Ionic equation: $Fe^{3+}(aq) + 3OH^{-}(aq) \rightarrow Fe(OH)_3(s)$ A brown/yellow ppt
Ag^{+}	Brown ppt soluble in excess giving colorless solution	Ag^{+} present, Ag_2O precipitated and dissolves forming $Ag(NH_3)_2^{+}$ ion.

SUMMARY

Add ammonia solution drop-wise until in excess

Observation	Deduction
• White precipitate soluble in excess	Zn^{2+} present
• White precipitate insoluble in excess	$Mg^{2+}, Pb^{2+}, Al^{3+}, Ba^{2+}$ or Sn^{2+} probably present
• No observable change	Ca^{2+} probably present
• White ppt insoluble in excess, darkens or turns brown on standing	Mn^{2+} present
• A pale blue precipitate soluble in	Cu^{2+} present

excess forming a deep blue solution	
• A dirty green precipitate insoluble in excess and turns brown on standing	Fe^{2+} present Fe^{2+} oxidized to Fe^{3+}
• A brown precipitate insoluble in excess	Fe^{3+} present
• A pale blue precipitate soluble in excess ammonia/ NH_4Cl solution and forms yellow- brown or red on standing	Co^{2+} present
• A green precipitate soluble in excess forming a pale blue solution	Ni^{2+} present
• Grey-green ppt insoluble in excess./ slightly soluble forming a pink or violet solution	Cr^{3+} present

7. ACTION OF DILUTE HYDROCHLORIC ACID OR DILUTE SULPHURIC ACID

Observation	Deduction
➤ No reaction in cold, bubbles of a colourless gas produced on warming. The colorless choking gas produced turns orange $K_2Cr_2O_7$ paper green and damp blue litmus red.	SO_2 gas produced SO_3^{2-} present
➤ No visible change/ reaction in cold and on warming.	SO_4^{2-} present
➤ Solution turns slightly cloudy white (Pale yellow) and denser on warming. The colourless choking gas produced turns orange $K_2Cr_2O_7$ paper green and damp blue litmus red.	SO_2 gas produced. Sulphur is slowly precipitated $S_2O_3^{2-}$ present
➤ No reaction in cold. Bubbles of a pale blue gas are produced on warming. The choking smell of the gas turns blue litmus red and bleaches it, KI solution turns brown.	Cl_2 gas produced. $S_2O_8^{2-}$ present ClO^- also liberates Cl_2 gas with a dilute acid
➤ Bubbles of a colourless gas which turn damp blue litmus pink(red) and lime water milky	CO_2 gas produced CO_3^{2-} or HCO_3^- present
➤ Colourless gas with rotten egg smell (poisonous) which turns damp blue litmus pink (red) and moist lead acetate paper black	H_2S produced S^{2-} present PbS formed
➤ Solution changes from yellow to	CrO_4^{2-} present

orange	CrO_4^{2-} turns to $\text{Cr}_2\text{O}_7^{2-}$
➤ Smell of vinegar	CH_3COO^- present
➤ White ppt soluble on warming and re-appears on cooling	Pb^{2+} present, PbCl_2 precipitated
➤ White ppt insoluble in excess acid and on warming. Dissolves in ammonia solution.	Ag^+ present. AgCl precipitated and dissolves in ammonia solution to give $\text{Ag}(\text{NH}_3)_2^+$ ion

NB: If no gas or vapour is produced but water insoluble solid dissolves in dilute sulphuric acid then metal oxides, OH^- present or salt of weak acid (stable and in volatile) i.e. OH^- , PO_4^{3-} , $\text{C}_2\text{O}_4^{2-}$, CrO_4^{2-} , O^{2-} present. Pb^{2+} , Ca^{2+} , Ba^{2+} are absent

8. ACTION OF CONC. SULPHURIC ACID ON SOLID SALTS OF INORGANIC SALTS

Observation	Deduction
✓ Yellow solid turns to dark/ bright red solid. Heat is evolved	CrO_4^{2-} present, CrO_3 formed. Exothermic reaction
✓ Orange solid substance turns to dark/ bright red solid. Heat is evolved	$\text{Cr}_2\text{O}_7^{2-}$ Present, CrO_3 formed. Exothermic reaction
✓ Colourless gas with rotten egg smell (Poisonous), turns damp blue litmus pink (red) and moist lead acetate paper black. Yellow white residue observed	H_2S produced S^{2-} present PbS and sulphur formed
✓ Colourless gas with choking smell, fumes heavily in air and forms dense white fumes with conc. Ammonia	Cl^- present Volatile HCl displaced
✓ On gentle warming colourless (pale brown) vapour slightly fumes in air. Colourless (pale yellow) liquid/condensate flows down the sides of the test tube. Brown fumes of pungent gas produced on continued heating	NO_3^- present Volatile HNO_3 displaced NO_2 gas produced
✓ Frothy effervescence of red- brown vapour, pungent, fuming heavily in air. Red- brown liquid condenses and runs back. Gas bleaches litmus	Br^- present Volatile HBr displaced Some oxidation to Br_2
✓ Frothy effervescence of red- brown vapours on warming. Red- oily liquid	$\text{Cr}_2\text{O}_7^{2-}$ and Cl^- (orange); CrO_4^{2-} and Cl^- (yellow) present. CrO_4^{2-} and Cl_2 formed, HCl

droplets condensing and running back. Heavy fuming in air	<i>produced.</i>
✓ Black solid precipitated, colourless pungent gas produced heavily in air. Violet vapour on heating, litmus paper bleached	<i>I⁻ Present, HI produced. Purple vapour is iodine (I₂)</i>
✓ Blue solid turns white	<i>Cu²⁺ present, formation of an hydrous sulphate from a hydrated salt</i>
✓ Red or pink solid turns blue	<i>Co²⁺ present, formation of an hydrous sulphate from hydrated salts</i>
✓ Dark brown solid yields a colourless gas that relights a glowing splint and leaves a white residue on heating	<i>PbO₂ present. Pb⁴⁺ converted to Pb²⁺ (PbSO₄), O₂ produced.</i>
✓ Black solid on heating liberates a colourless gas that re-lights a glowing splint. White solid left	<i>MnO₂ present MnO₂ changed to MnSO₄ MnO₂(s) + 2H₂SO₄(aq) → MnSO₄(aq) + O₂(g) + 2H₂O(l)</i>
✓ Orange-red solid yields a colourless odourless gas that re-lit a glowing splint	<i>Pb₃O₄ present. Pb₃O₄ reduced to PbSO₄</i>

9. REACTION WITH SODIUM CARBONATE SOLUTION.

TEST: add Na₂CO₃ solution drop wise to the solution, observe any change, then add reagent in excess. Warm and then boil with care

Observation	Deduction
White ppt, no other observable change	Pb ²⁺ , Zn ²⁺ , Ba ²⁺ , Ca ²⁺ , Mg ²⁺ present. PbCO ₃ , ZnCO ₃ , CaCO ₃ , BaCO ₃ , and MgCO ₃
White ppt, accompanied by effervescence of a colourless gas turns litmus red and lime water milky.	Very acidic solutions of Al ³⁺ , Sn ²⁺ present. Al(OH) ₃ pptd. Carbonates unstable hence CO ₂ produced.
White ppt (observe carefully), rapidly turning pale brown.	Mn ²⁺ present. MnCO ₃ ppt aerial oxidation to Mn ³⁺ compound.
Light blue ppt, darkening on heating and turning black.	Cu ²⁺ , CuCO ₃ ppt, decomposition to black CuO.
Light green ppt	Ni ²⁺ present, NiCO ₃ ppt.
Mud green ppt. (with Fe²⁺ the ppt may go through an initial white stage then darkness at the surface on	Fe ²⁺ , Cr ³⁺ present. FeCO ₃ and Cr ₂ (CO ₃) ₃ pptd, effervescence may occur with Cr ³⁺

standing)	
Red brown ppt accompanied by effervescence of a colourless gas, turns blue litmus red and lime water milky.	Acidic solution of Fe^{3+} present, $\text{Fe}(\text{OH})_3$ pptd. $\text{Fe}_2(\text{CO}_3)_3$, unstable hence CO_2 evolved.
Mauve ppt turning blue on heating	Co^{2+} present, CoCO_3 pptd.
Colourless, pungent gas turns red litmus blue evolved. Fumes with conc. HCl	NH_4^+ present. NH_3 evolved, Na_2CO_3 hydrolyses to give alkaline solution. $\text{CO}_3^{2-} + 2\text{H}_2\text{O} \longrightarrow \text{H}_2\text{CO}_3 + 2\text{OH}^-$ $\text{NH}_4^+ + \text{OH}^- \longrightarrow \text{NH}_3 + \text{H}_2\text{O}$
Effervescence of a colourless gas evolved, turning lime water milky. No other observable change.	Acid, or acid salt of strong acid e.g. HSO_4^- Present, CO_2 evolved
Colour of solution changes from orange to yellow	$\text{Cr}_2\text{O}_7^{2-}$ present. $\text{Cr}_2\text{O}_7^{2-}$ presence of OH^- Changes to CrO_4^{2-}

10. REACTIONS WITH SILVER NITRATE SOLUTION

Observation	Deduction
❖ White curdy ppt turning buff (flesh coloured) on heating.	CO_3^{2-} or HCO_3^- Present, Ag_2CO_3 pptd.
❖ White ppt turning purplish –grey on standing in bright light, insoluble in dil. HNO_3 but soluble in $\text{NH}_3(\text{aq})$	Cl^- probably present. AgCl pptd. AgCl forms soluble $\text{Ag}(\text{NH}_3)_2^+$ ion
❖ Pale cream ppt (sometimes almost white ppt), insoluble in dil. HNO_3 but soluble in conc. NH_3	Br^- present AgBr ppt
❖ Deep cream ppt (sometimes almost yellow) insoluble in both dil. HNO_3 and conc. NH_3	AgI pptd I^- Present
❖ Red ppt from yellow solution	CrO_4^{2-} present Ag_2CrO_4 pptd
❖ Red ppt from orange solution	$\text{Cr}_2\text{O}_7^{2-}$ Ag_2CrO_4 pptd.
❖ Black ppt	S^{2-} present, Ag_2S pptd
❖ Silver mirror (sometimes brownish ppt) observed on warming	Ag pptd, Fe^{2+} probably present i.e. $\text{Fe}^{2+} + \text{Ag}^+ \rightarrow \text{Fe}^{3+} + \text{Ag}(\text{s})$.

11. REACTIONS WITH LEAD (II) NITRATE SOLUTION

Test: Add lead (II) acetate or nitrate drop wise, then heat gently cool afterwards.

Observation	Deduction
○ White ppt, partially or completely soluble on heating, re-precipitating on cooling white crystals	Cl^- present

○ White ppt insoluble on heating; insoluble in dil HNO_3	SO_4^{2-} Present
○ White ppt insoluble on heating; ppt dissolves in dil. HNO_3 with effervescence of a colourless, odourless gas turns blue litmus red and lime water	CO_3^{2-} Present, PbCO_3 pptd CO_2 evolved
○ Yellow ppt, no visible color changes on heating. If ppt is small, it may dissolve on heating then re-precipitates on cooling.	I ⁻ present PbI_2 pptd
○ Yellow ppt, turning orange on heating	CrO_4^{2-} or $\text{Cr}_2\text{O}_7^{2-}$ present, PbCrO_4 pptd.
○ No apparent/ visible colour change	NO_3^- or CH_3COO^- present

12. REACTION WITH POTASSIUM IODIDE SOLUTION

Observation	Deduction
➤ Yellow ppt stained white (or cream ppt) in a brown (or red-brown) solution insoluble in excess reagent	Cu^{2+} present, CuI pptd. I_2 liberated $2\text{Cu}^{2+}(\text{aq}) + 4\text{I}^-(\text{aq}) \rightarrow \text{Cu}_2\text{I}_2(\text{s}) + \text{I}_2(\text{aq})$
➤ Yellow ppt insoluble in excess reagent. (if the amount of ppt is much and concentration of I ⁻ is low, then ppt will not dissolve in excess KI) ❖ Yellow ppt soluble in excess KI forming a colourless solution. (The ppt dissolves in conc. of I ⁻ is high and quantity of ppt is little)	Pb^{2+} present PbI_2 pptd Pb^{2+} present, PbI_2 pptd $(\text{PbI}_4)^{2-}$, soluble complex formed i.e. $\text{PbI}_2(\text{s}) + 2\text{I}^-(\text{aq}) \rightarrow \text{PbI}_4^{2-}(\text{aq})$
➤ Solution turns red/brown	Fe^{3+} , I_2 liberated

NOTE:

- Some oxidizing agents such as CrO_4^{2-} or $\text{Cr}_2\text{O}_7^{2-}$, IO_3^- can only liberate I_2 from KI in the presence of an acid. But Cu^{2+} and Fe^{3+} may not require an acid since the aqueous solutions are acidic
- Iodine may be liberated as a pale yellow or dark brown solution (if conc. is low) or as a black ppt) if conc. is high.
- Iodine liberated may be detected by:
 - Boiling the solution or suspension, forming purple vapor of I_2
 - Adding starch, forming deep blue colour
 - Adding CCl_4 and shaking, forming pink or purple lower layer

13. REACTIONS WITH POTASSIUM CHROMATE AND POTASSIUM DICHROMATE

Test: Add few drops of the reagent to the test solution; observe any changes taking place, the warm

Observation	Deduction
Pale yellow ppt, formed insoluble in NaOH(aq)	Ba ²⁺ present, BaCrO ₄ pptd
Yellow ppt, turns orange on heating, yellow ppt dissolves in NaOH(aq) forming a colourless solution	Pb ²⁺ present, PbCrO ₄ pptd. PbCrO ₄ dissolves to form, Pb(OH) ₄ ²⁻ ion
Yellow (CrO₄²⁻) solution turns green-blue or orange (Cr₂O₇²⁻) solution turns green Note: If I⁻ present, I₂ is liberated in the presence of H⁺, dark brown colour of I₂ will mask the green Cr³⁺, solid I₂ may be pptd.	Cr ⁶⁺ reduced to Cr ³⁺ reducing agent present in acid solution e.g. Sn ²⁺ also decolourizes MnO ₄ ⁻ /H ⁺ solution.

14. REACTIONS WITH HYDROGEN SULPHIDE GAS OR SODIUM SULPHIDE SOLUTION

Test: Add H₂S through a little of the test solution slowly, then moderately or add sodium sulphide solution to the test solution drop wise then in excess.

Observation	Deduction
a) A colourless solution giving dark brown-black ppt	Pb ²⁺ present from neutral acidified or alkaline solution. PbS pptd.
b) A colourless solution giving dark brown ppt	Sn ²⁺ present, from neutral acidified or alkaline solution.
c) A colourless solution giving white ppt (solution buffered with NH ₄ Cl/NH ₃)	Zn ²⁺ present, from only neutral acidified or alkaline solution not acidic. ZnS pptd, Zn ²⁺ present as Zn(NH ₃) ₄ ²⁺
d) A blue or green solution giving black ppt	Cu ²⁺ , CuS pptd from neutral, acidified or alkaline solution of Cu ²⁺
e) A yellow solution giving black ppt. buffered solution with NH ₄ Cl/NH ₃)	Fe ³⁺ from neutral, alkaline or acidified Fe ³⁺ ions H ₂ S oxidized to S and Fe ³⁺ reduced to Fe ²⁺
f) A blue solution giving black ppt. buffered solution with NH ₄ Cl/NH ₃)	Co ²⁺ present. CoS pptd. Co present as Co(NH ₃) ₄ ²⁺
g) Yellow solution giving muddy suspension (from neutral test	CrO ₄ ²⁻ , H ₂ S oxidized to S and Cr ⁶⁺ reduced to Cr ³⁺

solution)	
h) Orange solution giving muddy green suspension (from neutral test solution)	$\text{Cr}_2\text{O}_7^{2-}$, H_2S oxidized to S and Cr^{6+} reduced to Cr^{3+} .

15. CONFIRMATORY TESTS FOR CATIONS.

ION	TEST	OBSERVATION
Pb^{2+}	Heat suspected solid	Turns brown when hot and yellow when cold
	Add potassium iodide solution	A yellow precipitate $\text{Pb}^{2+}(\text{aq}) + 2\text{I}^{-}(\text{aq}) \rightarrow \text{PbI}_2(\text{s})$ A yellow ppt
	Add dilute hydrochloric acid solution	A white precipitate which dissolves on warming to give a colourless solution and reforms on cooling $\text{Pb}^{2+}(\text{aq}) + 2\text{Cl}^{-}(\text{aq}) \rightarrow \text{PbCl}_2(\text{s})$ A white ppt
	Add dilute sulphuric acid solution	A white ppt of PbSO_4 is formed
	Add potassium chromate solution	A yellow precipitate of lead chromate is formed $\text{Pb}^{2+}(\text{aq}) + \text{CrO}_4^{2-}(\text{aq}) \rightarrow \text{PbCrO}_4(\text{s})$ A yellow ppt
Cu^{2+}	Heat suspected solid	Turns black on heating

	Add sodium hydroxide solution drop-wise until in excess	A pale blue precipitate insoluble in excess. The ppt turns black on heating Ionic equation: $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ A pale blue ppt
	Add ammonia solution drop-wise until in excess	A pale blue precipitate soluble in excess to form a deep blue solution Ionic equation: $\text{Cu}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Cu}(\text{OH})_2(\text{s})$ A pale blue ppt $\text{Cu}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow \text{Cu}(\text{NH}_3)_4^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq})$ Tetra ammine copper(II) ions Deep blue solution
	Add potassium or sodium iodide solution	A white precipitate is formed in a brown solution $2\text{Cu}^{2+}(\text{aq}) + 4\text{I}^{-}(\text{aq}) \rightarrow \text{Cu}_2\text{I}_2(\text{s}) + \text{I}_2(\text{aq})$
	Add potassium thiocyanate solution	Reddish- brown precipitate
	Add potassium hexacyanoferrate (II) solution ($\text{K}_4\text{Fe}(\text{CN})_6$)	A reddish- brown gelatinous ppt of $\text{Cu}_2\text{Fe}(\text{CN})_6$ is formed
Zn^{2+}	Heat suspected solid	Turns yellow when hot and white when cold

	Add sodium hydroxide solution drop-wise until in excess	White precipitate soluble in excess to form a colourless solution Ionic equation: $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ White ppt $\text{Zn}(\text{OH})_2(\text{s}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Zn}(\text{OH})_4^{2-}(\text{aq})$ Zincate ion (colourless)
	Add ammonia solution drop-wise until in excess	White precipitate soluble in excess to form a colourless solution Ionic equation: $\text{Zn}^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq}) \rightarrow \text{Zn}(\text{OH})_2(\text{s})$ A white ppt $\text{Zn}(\text{OH})_2(\text{s}) + 4\text{NH}_3(\text{aq}) \rightarrow \text{Zn}(\text{NH}_3)_4^{2+}(\text{aq}) + 2\text{OH}^{-}(\text{aq})$ Tetra ammine Zinc(II) ions Colourless solution
	Add solid NH_4Cl followed by disodium hydrogen phosphate, then excess ammonia	White ppt of ZnNH_4PO_4 soluble in excess ammonia
	Add 1 or 2 drops of NH_3 (aq) (avoid excess). Decant, heat strongly till no further change	White ppt of $\text{Zn}(\text{OH})_2$ gives ZnO, which is yellow (hot) and white (cold)
	Add potassium Ferro-cyanide solution	A white precipitate soluble in alkalis
	Add NH_4Cl, then NH_3 (aq) then pass through H_2S or add Na_2S	White (dirty white) ppt of ZnS formed

NH_4^+	Add dilute sodium hydroxide and warm gently, test with a damp red and blue litmus paper	A colourless gas with a pungent choking smell that turns damp red litmus paper blue and forms dense white fumes with conc. HCl was produced.
	Heat the suspected solid	A white sublimate formed at the cooler parts of the test-tube
Fe^{2+}	Add potassium hexacyanoferrate(II) solution	A white precipitate turns to blue ppt
	Add $NaOH(aq)$ till in excess, leave to stand	Muddy green- gelatinous ppt of $Fe(OH)_2$ turns brown at the surface due to formation of $Fe(OH)_3$
	Add potassium hexacyanoferrate(III) solution	A dark or deep blue ppt of $Fe_3Fe(CN)_6$ is formed
	Add $1cm^3$ of H_2O_2, warm the add 3 drops of dil. HCl	Yellow/ brown solution is formed. $H_2O_2 + 2H^+ + 2Fe^{2+} \rightarrow 2H_2O + 2Fe^{3+}$
	Add dimethylglyoxime solution	Light red solution
Fe^{3+}	Add potassium hexacyanoferrate(II) solution	A deep blue ppt is formed
	Add potassium hexacyanoferrate(III) solution	Dark solution is formed
	Add potassium or ammonium thiocyanate, NH_4SCN (crystals or solution)	Deep red solution ($Fe(SCN)_6^{3-}$) is formed
	Add dimethylglyoxime solution	A red solution
	Add a few drops of H_2O_2, followed by dil. $NaOH$	Yellow or brown solution turns to green solution. Rapid effervescence of a gas that re-lit a glowing splint. $H_2O_2 + 2OH^- + 2Fe^{3+} \rightarrow 2H_2O + 2Fe^{2+} + O_2$

Ca²⁺	Flame test	Burns with a characteristic brick-red flame
	Add ammonium oxalate(or ethandioate), $(NH_4)_2C_2O_4$ followed by ethanoic acid and warm	White ppt of CaC_2O_4 formed which does not dissolve on warming with ethanoic acid
	Add potassium chromate solution	No precipitate formed
	Add dil. H_2SO_4 or Na_2SO_4 solution.	White ppt of $CaSO_4$ formed
	Add ammonium chloride followed by potassium Ferro cyanide	White ppt is formed
	Add $(NH_4)_2CO_3$ in the presence of NH_4Cl	White ppt of $CaCO_3$
Ba²⁺	Add potassium chromate solution	A pale yellow ppt of $BaCrO_4$
	Flame test: treat a little solution containing Ba²⁺ ions with one drop of conc. HCl, moisten the wire with the mixture, hold it in the edge of non-luminous flame	Burns with green(apple flame)
	Add saturated $CaSO_4$ solution	White ppt of $BaSO_4$
	Add dil. H_2SO_4 or Na_2SO_4 solution.	White ppt of $BaSO_4$ formed
	Add ammonium oxalate(or ethandioate), $(NH_4)_2C_2O_4$ followed by ethanoic acid and warm	White ppt of BaC_2O_4 formed which dissolves on warming with ethanoic acid
	Add $(NH_4)_2CO_3$ in the presence of NH_4Cl	White ppt of $CaCO_3$
Al³⁺	Add 1 or 2 drops of litmus solution, followed by dil. HCl, then finally add $NH_3(aq)$ until just alkaline	Blue lake formed

	Add disodium hydrogen phosphate solution	A white precipitate is formed soluble in mineral acids
	Add sodium carbonate solution	A white ppt of Aluminium hydroxide soluble in excess
	Add ammonia solution followed by a few drops of Alzarine solution	Pink solution
	To 3 cm³ of solution of Al³⁺ ions add 1 drop of cobalt (II) nitrate solution. Moisten a piece of filter paper with this mixture and heat the paper strongly by placing it on heated wire gauze or holding it directly on the flame	Bright blue ash of cobalt(II) aluminate is observed
Mg²⁺	Add a few crystals of <i>NH₄Cl</i> followed by 2-3 drops of disodium hydrogen phosphate (<i>Na₂HPO₄</i>) then finally add <i>NH₃(aq)</i> drop wise till in excess	White crystalline ppt, insoluble in <i>NH₃(aq)</i>.
	Add a few drops of Magneson followed by little sodium hydroxide solution	A blue ppt is formed
Ni²⁺	Add ammonia solution drop wise until in excess	Pale green ppt (use conc. solution) dissolves in excess giving a pale blue solution of <i>Ni(NH₃)₄²⁺</i>
	Add ammonia solution until the solution is just alkaline. Then add 2-3 drops of dimethyl-glyoxime solution	A pale green ppt forms red ppt of nickel(II)dimethyl-gyloxime complex
	Add potassium hexacyanoferrate(II) solution	A green precipitate soluble in ammonia solution $2Ni^{2+} + Fe(CN)_6^{4-} \rightarrow Ni_2Fe(CN)_6$
	Add potassium hexacyanoferrate(III) solution	A brown precipitate is formed $3Ni^{2+} + 2Fe(CN)_6^{3-} \rightarrow Ni_3Fe_2(CN)_{12}$

	Add 2-naphthol	Brown precipitate soluble in dilute hydrochloric acid
	Add potassium cyanide solution	A yellowish- green ppt of $\text{Ni}(\text{CN})_2$ which dissolves in excess forming a dark yellow solution of $\text{K}_2\text{Ni}(\text{CN})_4$
Mn^{2+}	Add a few drops of conc./ dil. HNO_3 followed by a little solid sodium bismuthate(V), BiO_3^-	Violet or purple colouration/ solution of MnO_4^- formed, may settle down as a dark brown ppt of MnO_2 $5\text{BiO}_3^- + 2\text{Mn}^{2+} + 14\text{H}^+ \rightarrow 2\text{MnO}_4^- + 5\text{Bi}^{3+} + 7\text{H}_2\text{O}$
	Add lead (IV) oxide followed by conc. HNO_3 and boil.	Colourless solution turns purple $5\text{PbO}_2 + 2\text{Mn}^{2+} + 4\text{H}^+ \rightarrow 2\text{MnO}_4^- + 5\text{Pb}^{2+} + 2\text{H}_2\text{O}$
	Fuse solid containing Mn^{2+} with a large excess fusion mixture ($\text{Na}_2\text{CO}_3 / \text{KNO}_3$)	Green mass of manganate(VI) MnO_4^{2-} is observed
	Add 1 cm³ of H_2O_2 then add 3 drops of dil. NaOH.	Dark brown ppt MnO_2 formed. Rapid effervescence of a colourless gas that re-lit a glowing splint. Much heat evolved
	Add $\text{NH}_4\text{Cl}/\text{NH}_3(\text{aq})$ till the solution is alkaline then pass H_2S or add Na_2S	Dirty white or pink ppt of MnS observed
Co^{2+}	Add 2-3 drops of ammonium thiocyanate (NH_4SCN) solution. Add some pentanol (amyl alcohol) or ether, shake gently.	Initial blue (pinkish- purple) solution of cobalt(II)thiocyanic acid, $\text{H}_2\text{Co}(\text{SCN})$ is observed, separates on shaking with amyl alcohol to form upper blue layer (in alcohol layer)
	Add potassium cyanide solution	Reddish brown precipitate soluble in excess $\text{Co}^{2+} + 2\text{CN}^- \rightarrow \text{Co}(\text{CN})_2$ $\text{Co}(\text{CN})_2 + 4\text{CN}^- \rightarrow \text{Co}(\text{CN})_4^{2-}$
	Bubble H_2S gas followed by dil. HCl	A black ppt insoluble in acid $\text{Co}^{2+} + \text{S}^{2-} \rightarrow \text{CoS}$

	Add Conc. HCl followed by water	Blue colour is formed which turns to pink on addition of water
	Add potassium thiocyanate solution	A blue solution is formed
Cr³⁺	- Add <i>NaOH(aq)</i> drop wise till in excess followed by a few drops of <i>H₂O₂</i> then heat to oxidize the chromite to yellow <i>CrO₄²⁻</i> then add: - Pentanol (amyl alcohol), then dil. <i>H₂SO₄</i>, shake gently - Lead(II) nitrate to the yellow solution (<i>CrO₄²⁻</i>)	- A green ppt soluble in excess forming a green solution. A yellow solution of <i>CrO₄²⁻</i> is formed $2Cr(OH)_3 + 3H_2O_2 + 4OH^- \rightarrow 2CrO_4^{2-} + 8H_2O$ - Blue colouration that concentrates in alcohol layer - Yellow ppt of <i>PbCrO₄</i> formed confirms Cr³⁺
	Fuse solid with large excess of fusion mixture (<i>Na₂CO₃/KNO₃</i>)	Yellow solid of chromate (<i>CrO₄²⁻</i>) obtained
Sn²⁺	Add <i>Na₂S</i> solution	Brown ppt of <i>SnS</i>
	Add potassium manganate(VII) solution	Purple color of <i>MnO₄⁻</i> turns colourless
	Add potassium chromate (VI) solution	Yellow colour of <i>CrO₄²⁻</i> turns green-blue due to formation of <i>Cr³⁺</i>
	Add iron(III) chloride solution	Yellow- brown, <i>Fe³⁺</i> solution turns to pale green colour of <i>Fe²⁺</i>

16.DETECTION OF ANIONS

Dissolve a little of the substance in cold water or to a solid substance, then carry out the identification test for the anion

ION	TEST	OBSERVATION
CO_3^{2-} or HCO_3^-	Heat the solid except Na_2CO_3 and K_2CO_3	Effervescence of a colourless gas which turns lime water milky and damp blue litmus red
	To the solid or solution add dilute acid	Effervescence of a colourless gas (CO_2 gas) which turns lime water milky and damp blue litmus red
	Differentiating between CO_3^{2-} and HCO_3^-. All HCO_3^- are soluble in water and only Na^+, K^+ and NH_4^+ bicarbonates are available in solid form.	
	a) Add 1-2 drops of $MgSO_4(aq)$	No ppt, formation of soluble $Mg(HCO_3)_2$ confirms HCO_3^- .
	b) Add lead(II)acetate/nitrate, heat then add dil. HNO_3	White ppt of $PbCO_3$ insoluble on heating but soluble in dil. HNO_3 with effervescence of a colorless gas that turns damp blue litmus red and lime water milky
SO_4^{2-}	Add dil. HCl followed by a few drops of $BaCl_2(aq)$	White ppt of $BaSO_4$
	Add dil. HNO_3 followed by a few drops of $Ba(NO_3)_2(aq)$	White ppt of $BaSO_4$
	Differentiating between SO_4^{2-} and HSO_4^- (all HSO_4^- are soluble in water and only Na^+, K^+ and NH_4^+ hydrogen sulphates are ordinarily available.	
	Heat the solid gently	Dense white choking fumes, turn blue litmus red and $Ba(NO_3)_2(aq)$ milky. If SO_3 is readily evolved then HSO_4^- is confirmed
		If SO_3 fumes are evolved only on strong heating then SO_4^{2-} is confirmed

	Add solid Na_2CO_3 powder	Vigorous effervescence of CO_2 confirms HSO_4^-
		If there is slight or no effervescence at all, then SO_4^{2-} is confirmed.
SO_3^{2-}	Add dil. HCl and warm	No reaction in cold, bubbles of a colourless gas produced on warming. The colourless choking gas (SO_2) produced turns orange $\text{K}_2\text{Cr}_2\text{O}_7$ paper green and damp blue litmus red and bleaches it.
	Add iodine solution	The brown color is immediately decolourized
	Add $\text{FeCl}_{3(aq)}$ acidify, warm and add $\text{NaOH}_{(aq)}$	A dark red- brown solution observed becomes almost colourless when hot. Green ppt formed with $\text{NaOH}_{(aq)}$. Fe^{3+} reduced to Fe^{2+}
	Add 2-3 drops of barium nitrate solution followed by dilute nitric acid	White ppt soluble in dilute nitric acid
$\text{S}_2\text{O}_3^{2-}$	Add dil. HCl and warm	White (or cream) ppt of sulphur with evolution of bubbles of a colourless gas produced on warming. The colourless choking gas (SO_2) produced turns orange $\text{K}_2\text{Cr}_2\text{O}_7$ paper green and damp blue litmus red.
	Add $\text{I}_2(aq)$ in KI solution	The brown color of I_2 was immediately decolorized $\text{I}_2 + 2\text{S}_2\text{O}_3^{2-} \rightarrow 2\text{I}^- + \text{S}_4\text{O}_6^{2-}$
	Add silver nitrate solution	Yellow ppt that turns black
	Add $\text{FeCl}_{3(aq)}$ acidify, warm and add $\text{NaOH}_{(aq)}$	Dark purple solution clears when hot, becomes cloudy. Green ppt of $\text{Fe}(\text{OH})_2$ with $\text{NaOH}_{(aq)}$ $2\text{Fe}^{3+} + 2\text{S}_2\text{O}_3^{2-} \rightarrow 2\text{Fe}^{2+} + \text{S}_4\text{O}_6^{2-}$

NO_3^-	To a solid or solution add a few pieces of copper turnings, then about 2cm^3 of conc. H_2SO_4. Heat gently NB: NO_2^- gives immediate effervescence before warming	Brown fumes of gas and a blue solution (Cu^{2+} ions) on heating confirms NO_3^-
	To a solid or solution add a few drops of NaOH(aq) then a little of zinc or aluminum powder or Devarda's alloy and heat the mixture	Evolution of a colourless pungent choking gas, turns red litmus blue and fumes heavily with conc. HCl (NH_3 gas produced) confirms NO_3^-
	Brown ring test: To the test solution add an equal volume of cold freshly prepared $\text{FeSO}_{4(\text{aq})}$ solution followed by drops of conc. H_2SO_4. NB: I^-, Br^-, NO_2^- also form brown rings. If Pb^{2+}, Cu^{2+}, Ba^{2+} white ppt is formed but the brown ring is unaffected	Formation of a brown ring Fe(NO)^{2+} at the interface or aqueous layer- acid junction
NO_2^-	To solid or conc. solution add dil. HCl or H_2SO_4	Immediate effervescence of brown fumes (NO_2). Pale blue solution formed (i.e. HNO_2 or N_2O_3)
	Add fresh $\text{FeSO}_{4(\text{aq})}$ followed by dil. NaOH	Mixture turns black, $\text{Fe(H}_2\text{O)}_5(\text{NO})^{2+}$, then gave a yellow solution (Fe^{3+}) that gave red-brown ppt of Fe(OH)_3 confirms NO_2^-
	Add KMnO_4 solution	Solution becomes colourless or traces of brown ppt (MnO_2) which clear on standing
	To cold test solution add $\text{FeSO}_{4(\text{aq})}$ followed by dil. H_2SO_4	Dark brown complex ion, Fe(NO)^{2+} formed
$\text{C}_2\text{O}_4^{2-}$	Add 2-3 drops of AgNO_3 (aq) followed by excess NH_3 (aq).	White ppt ($\text{Ag}_2\text{C}_2\text{O}_4$) soluble in excess NH_3 (aq), forming a colourless complex $\text{Ag(NH}_3)_2^+$

	Add Barium chloride solution followed by dilute hydrochloric acid	White ppt dissolves without effervescence.
	Add a few drops of dil. H_2SO_4, heat the mixture to about $70^\circ C$ then add a few drops of $KMnO_4(aq)$	Purple solution turns colourless with bubbles of a colourless gas that turns lime water milky
CrO_4^{2-}	Add dil. H_2SO_4	Colour changes from yellow to orange
$Cr_2O_7^{2-}$	Add dil. $NaOH$ solution	Colour changes from orange to yellow
	Reactions in which CrO_4^{2-} resembles $Cr_2O_7^{2-}$	
	Add lead(II)acetate	Yellow ppt of $PbCrO_4$ formed
	Add $AgNO_3(aq)$	Brick-red ppt of Ag_2CrO_4
	Add pentanol and dil. H_2SO_4	Blue CrO_5 formed, more concentrated in the alcohol layer
S^{2-}	- To a test solid or solution add dil. HCl, warm - To a test solid or solution add dil. H_2SO_4, warm	Colourless gas with rotten egg smell, (H_2S) (poisonous) which turns damp blue litmus pink (red) and moist lead acetate paper black (PbS)
CH_3COO^-	To test or solution add dil. H_2SO_4, warm	Smell of vinegar from displaced CH_3COO^-
	Moisten a little test solid with ethanol then add a little conc. H_2SO_4 and pour into water. Smell	A sweet fruity smell of an ester formed from a salt of a carboxylic acid
Cl^-	Add 2-3 drops of $AgNO_3$, then a few drops of dil. HNO_3	White ppt of $AgCl$ insoluble in dilute acid but soluble in ammonia solution
	To a little solid add a few drops of conc. H_2SO_4 and warm	Colourless pungent fumes, that turn damp blue litmus red, fumes heavily in air or with a drop of conc. NH_3 i.e HCl produced
	To a little solid test substance add a little of manganese(IV)oxide followed by a few drops of conc. H_2SO_4 and warm	Pale green gas (yellow-green) bleaches damp blue litmus paper. Cl_2

	To a test solution add 1-2 drops of chlorine water(or slightly acidified $NaOCl$, sodium hypochlorite),then followed by 2-3 cm³ of CCl_4, shake	Colourless(or very pale green) lower layer confirms Cl^- $ClO^- + 2H^+ + Cl^- \rightarrow H_2O + Cl_2$
Br^-	Add 2-3 drops of $AgNO_3$, then a few drops of dil. HNO_3	Pale cream(pale yellow) ppt of $AgBr$ insoluble in dilute acid but soluble in ammonia solution
	To a little solid add a few drops of conc. H_2SO_4 and warm	Mixture of colourless pungent fumes of HBr and red-brown pungent, Br_2 gas condensed to brown liquid produced
	To a test solution add 1-2 drops of chlorine water(or slightly acidified $NaOCl$, sodium hypochlorite),then followed by 2-3 cm³ of CCl_4, shake	Yellow orange lower aqueous layer. Br^- oxidized to Br_2, layer decolorizes on addition to $NaOH(aq)$
	To a little solid test substance add a little of manganese(IV)oxide followed by a few drops of conc. H_2SO_4 and warm	Red-brown pungent vapours bleaches damp litmus paper condenses to brown liquid and forms brown solution with CCl_4, Br_2 produced
I^-	Add 2-3 drops of $AgNO_3$, then a few drops of dil. HNO_3	Yellow (or cream) ppt of AgI insoluble in both dilute acid and in ammonia solution
	To a little solid add a few drops of conc. H_2SO_4 and warm	Black ppt of I_2 formed, fuming $HI(g)$ also produced
	To a test solution add 1-2 drops of chlorine water(or slightly acidified $NaOCl$, sodium hypochlorite),then followed by 2-3 cm³ of CCl_4, shake	Violet/ purple pungent vapour slowly bleaches damp litmus and forms blue colouration with starch, I_2 produced
	To a little solid test substance add a little of manganese(IV)oxide followed by a few drops of conc. H_2SO_4 and warm	Pink or purple lower layer confirms I^-, I^- oxidized to I_2

ORGANIC QUALITATIVE ANALYSIS

Qualitative analysis deals with identification of the nature or the functional groups (reactive centers) present in an organic compound. Functional groups to be analyzed mainly include

- Hydroxyl (-OH) group; for alcohols and phenols
- Carbonyl group(-C=O), for carbonyl compounds (aldehydes and ketones)
- Carboxyl group (-COOH) for carboxylic acids
- Amino group(-NH₂),for amines
- Others could be; ethers(-O-), alkyl and aryl halides(R-X and Ar-x), esters, amines

PRELIMINARY TESTS

1. APPEARANCE AT 20°C (room temperature)

- A majority of lower aliphatic compounds such as alcohols, aldehydes, ketones, esters, ethers, amines etc are liquids at 20° C
- Most aromatic compounds are solids at room temperature. Some exceptions such as phenylamine, benzaldehyde, benzene, methylbenzene, nitrobenzene, halogenobenzenes, benzyl chloride, phenyl methanol(benzyl alcohol)

NB; There are no gaseous aromatic compounds at 20°C

2. ODOURS/ SMELL

Students should familiarize themselves with the following characteristic odours which may be difficult to describe in words

Odour/ smell	Class of compounds
Pleasant, fruity	Esters
Pungent	Lower acid chlorides, acid anhydrides, aldehydes
Almond	Benzaldehydes, nitro- compounds
Carbolic	Phenols
Antiseptic	Triiodomethane, halogenated phenols
Odourless	Ionic compounds, high molecular weight compounds
Petrol or paraffin	Liquid alkanes

3. COLOURS

A great majority of organic compounds are colourless. Some simple coloured organic compounds include;

- Azo- dyes i.e. 2,4-dinitrophenylhydrazones are red or orange
- Triiodomethane, nitro- compounds are yellow
- Many metallic salts of organic acids are coloured

4. SOLUBILITY OF ORGANIC COMPOUNDS IN WATER

Solubility of organic compounds in water is related to:

- The functional group present, functional groups containing more electronegative elements such as oxygen and nitrogen are highly polar and the extent to be soluble in water due to hydrogen bonding occurs
- The size of the hydrocarbon skeleton (the larger the non-polar part of the organic molecule the less soluble it is)

Soluble(lower aliphatic cpds)	Sparingly soluble
Acids	Aromatic compounds
Alcohols	Simple phenols
Aldehydes	Esters
Amines	Ethers
Salts of Na^+ , K^+ , NH_4^+	Halogenoalkanes

Test: To about 0.5g or 1cm³ of organic substance in a test-tube, add 1or 2 drops of distilled water. Observe, and then add about 3cm³ of more distilled water. Note the solubility in cold water. Warm the mixture and note any change.

Observation	Deduction
a) Substance readily soluble. No hydrogen chloride gas produced Red litmus turns blue	Alkaline solution, Amine present $RNH_2 + H_2O \leftrightarrow RNH_3^+ + OH^-$ Salt of a weak acid and strong base hydrolyzed present $CH_3COONa + H_2O \leftrightarrow CH_3COOH + NaOH$
Blue litmus turns red	Carboxylic or sulphonic acid present, Phenol present, Salt of a strong acid and a weak base present which is hydrolyzed present $RNH_3^+ + H_2O \leftrightarrow H_3O^+ + RNH_2$

• Neutral to litmus	Neutral substance present e.g. alcohol, aldehydes and ketones, esters, amides
b) Substance forms a separate layer (observe carefully) soluble on standing, shaking or on gentle warming to give an acidic solution. No <i>HCl</i> gas produced	Phenol present, acid anhydride present Acid formed from hydrolysis
c) Hydrogen chloride gas produced, the substance dissolves giving an acidic solution • Rapidly or vigorously to give a colourless solution • Gradually on boiling to yield a white ppt (immediately or on cooling)	Acid(acyl) chloride present, which is hydrolyzed • Lower aliphatic acid chloride present yields water soluble acid.(these substances fume in air and are extremely pungent) • Aromatic acid present, yield an acid which is sparingly soluble in cold water

5. COMBUSTION / EFFECT OF HEAT

TEST: Burn a small amount of the substance on a crucible lid

Observation	Deduction
• Substance burns readily with a blue, non-sooty, non-luminous flame	Saturated aliphatic compound with low C: H ratio possibly not more than 4 or 5 carbon atoms per molecule
• Substance burns readily with a yellow, slightly sooty, luminous flame	Unsaturated aliphatic compound
• Substance burns readily with a yellow, heavily sooty (smoky), luminous flame. Black solid (carbon) deposited on cold parts of spatula or glass rod held at the top of the flame	High C: H ratio probably aromatic or highly unsaturated aliphatic or highly aliphatic

N.B; (EFFECT OF HEAT)

If a substance is in a solid form, put it in a dry glass test tube and heat first gently, then more strongly. Smell any possible gaseous products with care.

Observation	Deduction
➤ Substance melts into a solution (in its own water of crystallization). CO and CO ₂ evolved with some charring. CO burns in tube (delivery tube) with quite blue flame if ignited. A colourless condensate (water) is observed.	Ethanedioic (oxalic) acid, or ethanedioate (oxalate) present $(\text{COOH})_2 \longrightarrow \text{CO} + \text{CO}_2 + \text{H}_2\text{O}$.
➤ H ₂ and CO evolved in two stage reaction. H ₂ burns if ignited, on strong heating CO is evolved.	Methanoate (formate) present $2\text{H COONa} \longrightarrow (\text{COONa})_2 + \text{H}_2$ $(\text{COONa})_2 \longrightarrow \text{Na}_2\text{CO}_3 + \text{CO}$
➤ Ketone evolved (characteristics odour), vapour burns with luminous flame if ignited	Metal salt of a carboxylic acid $2\text{RCOONa} \longrightarrow \text{Na}_2\text{CO}_3 + \text{RCOR}$
➤ Vapour non-flammable	Highly halogenated compounds e.g. CCl ₄ CHCl ₃
➤ Ammonia evolved (smell), sublimation occurs	Ammonium salt present or odour of acid detected

6. REACTION WITH SODIUM HYDROXIDE SOLUTION

TEST: To about 0.5g or 1cm³ of the substance in a test-tube, add about 5cm³ of 2M NaOH solution drop wise, shaking after each addition. Observe and note any gases produced. Warm and then boil. Test the gases or vapours evolved with moist red litmus paper

Observation	Deduction
1) Colourless original substance yields a yellow brown resin, which on boiling, precipitates with unpleasant smell	Aliphatic aldehyde present(not methanol); polymerization occurs
2) Substance dissolves a. Readily (though sparingly soluble in water) to give a colourless solution, no gas or vapour formed	Substance is acidic. Carboxylic acid or phenol present. Neutralization reaction occurs $\text{RCOOH} + \text{OH}^- \longrightarrow \text{RCOO}^- + \text{H}_2\text{O}$

b. Slowly on boiling	Hydrolysis to soluble products - Benzaldehyde and other aromatic aldehydes undergo disproportionation reaction i.e. $2C_6H_5CHO + OH^- \longrightarrow C_6H_5COO^- + C_6H_5CH_2OH$ - Esters and acid anhydrides also undergo hydrolysis
3) Strongly alkaline gas produced (red litmus turns blue) in cold or gentle warming NH_3 gas produced, gas does not burn	Displacement of a volatile weak base, NH_4^+ salt present
Amine evolved, fishy ammoniacal odour, vapour burns	Aliphatic amine salt present $RNH_3^+ + OH^- \longrightarrow RNH_2 + H_2O$
4) Yellow original substance dissolves to give an orange solution	A nitro phenol

7. REACTION WITH SODIUM CARBONATE SOLUTION

TEST: To about 0.5g or 1cm³ of the substance in a test-tube, add about 5cm³ of 2M Na_2CO_3 solution drop wise, Observe carefully, then warm gently and finally boil. Test for carbon dioxide using lime water, shaking after each addition. Observe and note any other gases produced.

Observation	Deduction
Yellow brown resin precipitated on boiling; unpleasant odour.	Aliphatic aldehydes present (but not methanal), polymerization reaction occurs.
Substance dissolves without effervescence, no other apparent / observable change.	Substance neutral or less acidic than carbonic acid present e.g. phenol, alcohols
- Effervescence i. CO_2 evolved, no other gas or vapour	Substance is more strongly acidic than carbonic acid, i.e. $CO_3^{2-} + 2H^+ \longrightarrow CO_2 + H_2O$ Substances possibly present are; Carboxylic acid
	Substituted phenol e.g. nitro phenols (these finally give a yellow solution)
	Sulphonic acid
ii. CO_2 and amine vapour evolved, fishy ammoniacal odour, turns moist red litmus blue.	Amine salt present, amine is volatile. Acid is strong. The amine salt solution is acidic by hydrolysis. $RNH_3^+ \rightleftharpoons RNH_2 + H^+$

CO₂ evolved and an oil separates	Amine salt present, amine is liquid at room temperature and sparingly soluble in water; parent acid is strong e.g. C ₆ H ₅ NH ₃ ⁺ Cl ⁻
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Note: Na₂CO₃ solution largely behaves like NaOH solution; due to hydrolysis of the CO₃²⁻ i.e



8. REACTION WITH DILUTE HYDROCHLORIC ACID ORSULPHURIC ACID

Test: to a little of the substance in a test tube, add 5cm³ of the dilute acid. Warm gently and finally boil.

Observation	Deduction
Substance dissolves readily in cold, no gas or vapour formed (though sparingly soluble in water). NB. Both aromatic and aliphatic amines dissolve exothermically, forming dense white fumes and a clear solution with HCl	Substance is basic, amine present; $RNH_2 + H^+ \longrightarrow RNH_3^+$ $R_2NH + H^+ \longrightarrow R_2NH_2^+$ $R_3N + H^+ \longrightarrow R_3NH^+$

9. REACTION WITH CONCENTRATED SULPHURIC ACID

Precaution! Test in this case should be performed with great care since conc. H₂SO₄ is potentially dangerous. A test-tube holder **must** be used, allow the test tube to cool completely before disposing the contents in to a sink of running water.

Test: Carefully add 1 or 2 drops of the acid to about 0.2g or 1 cm³ of the test substance. Add more (1 cm³) acid, observe carefully in cold the warm gently and observe again. Do not over heat (because choking white fumes may be observed)

Observation	Deduction
- Effervescence, cold or on gentle warming. Little or no blackening ➤ CO produced (no CO₂), burns with blue flame on ignition.	Methanoic (formic acid) acid or methanoate (formate) present. Dehydration occurs i.e. $HCOOH \longrightarrow CO + H_2O$
➤ CO and CO₂ produced	Ethanedioc(oxalic) acid or its salt present, Dehydration $(COOH)_2 \longrightarrow CO + CO_2 + H_2O$

Mixture in the tube solidifies, highly exothermic reaction	Amine present, salt formed i.e. $RNH_2 + H_2SO_4 \longrightarrow RNH_3^+ + HSO_4^-$
Some blackening on warming, no effervescence	Substance present are possibly Phenol Phenolic acid e.g. 2- hydroxyl benzoic acid, naphthalene -1-ol or salts
Liquid substance slowly dissolves; exothermic reaction, no other visible change.	An alkene probably present. Addition reaction occurs e.g. $RCH=CHR + H_2SO_4 \longrightarrow RCH_2CH(SO_3OH)R$

10. REACTION WITH BROMINE WATER (Br_2/CCl_4), SATURATED AQUEOUS SOLUTION OF BROMINE

TEST: To 0.5g or 1 cm³ of the test substance, drop wise add bromine water, shake after each addition. If decolourization (or partial decolorization or disappearance of brown colour occurs, continue adding bromine water until in excess.

Observation	Deduction
➤ Immediate decolourization of bromine, heavy white fumes formed	Amine present, white fumes are HBr formed
• Products completely miscible with water	Aliphatic amine present or aromatic amine with $-NH_2$ not attached to the benzene ring e.g. $C_6H_5CH_2NH_2$
• Products immiscible with water, white ppt on addition of excess bromine water	Aromatic amine present e.g. $C_6H_5NH_2 + 3Br_2 \longrightarrow C_6H_2Br_3NH_3 + 3HBr$
➤ Immediate decolourization of bromine and white fumes formed Products immiscible with water • White ppt with excess bromine water	Phenol present $C_6H_5OH + 3Br_2 \longrightarrow C_6H_2Br_3OH + 3HBr$
• Second liquid layer (excess bromine soluble in this layer)	NB; fumes of HBr rarely because of its high solubility in water An alkene or alkyne present; addition reaction
➤ Slow decolourization of bromine water (more rapid if acid is added), no white fumes	Aldehydes or ketone present. (Occasionally, a primary or secondary alcohol). Substitution reaction catalyzed by H^+ occurs e.g. $CH_3COCH_3 + Br_2 \longrightarrow CH_2BrCOCH_3 + HBr$

11. REACTION WITH IODINE AND SODIUM HYDROXIDE SOLUTION (IODO FORM REACTION)

TEST: To about 0.5cm³ of the test substance add about 4cm³ of iodine in potassium iodide solution. Then add 2M *NaOH* solution drop wise until the colour of iodine is first discharged, warm and cool.

OBSERVATION	DEDUCTION
Yellow ppt (in cold or gentle heating): antiseptic smell observed	Tri-iodomethane or iodoform, CHI_3 produced. Test substance contains either of the following structural groups $CH_3C=O$, CH_3C-O e.g. - Ethanal - Ethanol - Methyl ketone

NB: Similar reactions occur with $Br_{2(aq)}/NaOH_{(aq)}$, $Cl_{2(aq)}/NaOH_{(aq)}$ or $NaOCl$. But trichloromethane (chloroform), $CHCl_3$ are colourless liquids

Test	Observation	Deduction
To 1 cm³ of the test solution add 3 cm³ of $KI_{(aq)}$, then 10 cm³ of sodium chlorate(I), (sodium hypochlorite)	Yellow ppt	Tri-iodomethane, CHI_3 produced $CH_3C=O$ or CH_3CHOH present.

12. REACTION WITH 2,4-DINITROPHENYLHYDRAZINE (BRADY'S REAGENT)

This reagent is used to test for carbonyl group ($-C=O$) in aldehydes and ketones

Test	Observation	Deduction
If the test substance is a solid dissolve about 0.5g in methanol. To 1 cm³ of the reagent add several drops of the test substance (or its solution in methanol)	Yellow or yellow-orange ppt	Condensation reaction, aldehydes or ketone present

13. FEHLINGS' SOLUTION

This reagent is used to test for aldehydes and reducing sugars

Test	Observation	Deduction
To 1 g or 1 cm ³ of the test substance add about 6 cm ³ of Fehling's solution, warm and boil for 2-3 minutes	Red (or light brown) ppt on boiling. The ppt may start to appear as muddy yellow colour, then changes to red on heating	Copper (I) oxide formed. A reducing agent, possibly an aliphatic aldehydes present. C_6H_5CHO does not reduce Fehling's solution

14. REACTION WITH 0.1M COPPER (II) SULPHATE SOLUTION

Test	Observation	Deduction
To about 5cm³ of the test substance add 1cm³ of copper(II) sulphate solution	- Bright green colour which first darkens, then gives a yellow-green ppt - Blue-green ppt, which darkens and then dissolves to give a deep blue solution	Aromatic amine e.g. phenyl amine present Aliphatic amine e.g. butyl amine present

15. REACTION WITH AMMONIACAL SOLUTION OF SILVER NITRATE (Tollen's reagent)

Preparation

To 5cm³ of 0.1M silver nitrate solution add a few drops, about 4 of 2.0 M sodium hydroxide solution. A grey-brown ppt is formed. Then add 2M ammonia solution drop wise till the ppt just dissolves.

TEST: To the reagent prepared above add about 5cm³ of test substance (liquid or aqueous) and place the test tube in a beaker of hot water for a few minutes (or warm)

Observation	Deduction
Grey or dark grey ppt on the inner surface of test-tube	Metallic silver is produced by reducing agent - An aldehyde present - Methanoic acid (formic acid) or its salts. (reaction is very slow) NB: Redox reaction taking place $Ag(NH_3)_2^+ + e^- \longrightarrow Ag + 2NH_3$

16. REACTION WITH NEUTRAL IRON(III)CHLORIDE

Test	Observation	Deduction
To 1g(or 1 cm ³) of the test substance add neutral iron(III)chloride solution until no further change	• Violet colouration	• Phenol or phenolic derivative present
	• Brown solution	• $HCOO^-$, CH_3COO^- present
	• Green solution	• $C_2O_4^{2-}$ present

17. REACTION WITH ACIDIFIED POTASSIUM DICHROMATE SOLUTION

Test	Observation	Deduction
To 1cm ³ of sample, add 3-4 drops of acidified dichromate solution and heat gently	• Orange solution rapidly turned blue-green	Primary alcohol present Aldehydes present
	• Orange solution slowly changed blue-green	Secondary alcohol, aldehydes or methanoic acid present
	• No observable change	Tertiary alcohol or ketone present

18. REACTION WITH ACIDIFIED POTASSIUM MANGANATE (VII) SOLUTION

Test	Observation	Deduction
To 1cm ³ of test sample add 2-3 drops of acidified permanganate and warm/ heat gently	Purple colour turned colourless	Primary and secondary alcohol; aldehydes and methanoic acid present
		Alkenes and alkynes also decolourize permanganate solution

19. LUCAS REAGENT(solution of anhydrous zinc chloride in concentrated hydrochloric acid)

This test is used to distinguish between the classes of alcohols i.e. primary, secondary and tertiary

Test: To about 0.5 cm³ of an alcohol in a test tube, quickly add 2cm³ of Lucas reagent. Close the test-tube with a cork and shake vigorously and allow the mixture to stand in ice-cold water. Observe the mixture over time.

Observation	Deduction
No observable change at room temperature	Primary alcohol present
Solution turns cloudy after about 5-minutes (distinct upper layer may form after 1 hr.)	Alkyl chloride formed, secondary alcohol present
Solution turns cloudy immediately, clears and separates rapidly into two layers in about 1-2 minutes	Insoluble alkyl chloride formed. Tertiary alcohol present

20. REACTION WITH SODIUM NITRITE IN HYDROCHLORIC ACID,(Nitrous acid or Nitric(III)acid below 5°C

TEST; To 0.5g (or 0.5 cm³) of the test substance in a test-tube, add some water (or dilute *HCl*) and shake to dissolve. Place the tube in an ice- cold water bath. Add 2cm³ of sodium nitrite solution followed by 2cm³ of hydrochloric acid solution added drop wise. If no effervescence, warm gently.

Observation	Deduction
Clear solution with effervescence (bubbling) gas has no effect on lime-water and puts out a lighted splint	Nitrogen produced. Amino (-NH ₂) group present, not attached to the aromatic/benzene ring Primary aliphatic amine present $R-NH_2 + HNO_2 \longrightarrow ROH + N_2 + H_2O$
Yellow oil separates (occasionally, yellow solution or an emulsion is formed)	Secondary amine present, the yellow oily liquid is a nitroso-amine $R_2NH + HNO_2 \longrightarrow R_2NNO + H_2O$

No observable change in the cold, apart from slight decomposition of nitrous acid on warming, a black oil separates and phenolic (carbolic) odour produced and nitrogen produced	Primary aromatic amine present, a diazonium salt is produced i.e. $C_6H_5NH_2 + HNO_2 + H^+ \longrightarrow C_6H_5N_2^+ + 2H_2O$ On warming phenol and N_2 re produced i.e. $C_6H_5N_2^+ + H_2O \longrightarrow C_6H_5OH + N_2 + H^+$
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N.B: Nitroso amines are highly toxic; care should be taken to avoid contamination of the skin

21. AZO- DYE REACTION

TEST: To the test substance dissolve in dilute; add nitrous acid followed by an alkaline solution of 2-naphthol or naphthalene-2-ol or using phenol

Test	Observation	Deduction
Using HNO_2 and phenol	- Bright yellow ppt - Yellow solution	- Primary aromatic amine e.g. $C_6H_5NH_2$ present. - Primary aliphatic amine e.g. butyl amine present
Using HNO_2 and Naphthalene-2-ol	- Red/orange crystalline ppt - Yellow solution	- Primary aromatic amine present - Primary aliphatic amines