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 UNKNOWNS

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 ($CaOCl_2$**)**

NB:

- If the substance is a **crystalline solid** then; $\boldsymbol{Cl^-}, \boldsymbol{SO_4^{2-}}, \boldsymbol{NO_3^-}, \boldsymbol{C_2O_4^{2-}}, \boldsymbol{S_2O_3^{2-}}$ 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 $\boldsymbol{HCO_3^-}$**,** $\boldsymbol{OH^-}$ or $\boldsymbol{HSO_4^-}$**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 chocking 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, $O^{2-}, ClO_3^-, \boldsymbol{NO_3^-}$, 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, $\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$**or** $\boldsymbol{C_2O_4^{2-}}$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	$\boldsymbol{SO_3^{2-}}$ present $\boldsymbol{SO_3^{2-}}$ 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.	$\boldsymbol{S_2O_3^{2-}}$ present $\boldsymbol{S_2O_3^{2-}}$ decomposed to sulphur (yellow) which melts to thick black liquid.
xi.	Yellow residue formed, turned brown and gave a black viscous liquid	$\boldsymbol{S_2O_3^{2-}}$ present $\boldsymbol{S_2O_3^{2-}}$ 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, $\boldsymbol{Cu^{2+}}$ **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 $\boldsymbol{SO_4^{2-}}$ 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.	$\boldsymbol{Fe^{3+}}$ **present**
iii. Green solid dissolves forming a green solution.	Hydrated salt of $\boldsymbol{Fe^{2+}}$, $\boldsymbol{Cu^{2+}}$ $\boldsymbol{Ni^{2+}}$or $\boldsymbol{Cr^{3+}}$ probably present
iv. Blue solid dissolves giving a blue solution.	Hydrated salt of $\boldsymbol{Cu^{2+}}$ $\boldsymbol{Ni^{2+}}$, $\boldsymbol{Cr^{3+}}$or $\boldsymbol{Fe^{2+}}$probably present.
v. Yellow solid dissolves giving a yellow solution	$\boldsymbol{Fe^{3+}}$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	$\boldsymbol{Mn^{7+}}$(from a permanganate) or $\boldsymbol{Cr^{3+}}$ **(**from conc. Solutions)
viii. Pink or red solid dissolves giving pink or	Hydrated $\boldsymbol{Co^{2+}}$ **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 in**soluble** 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 chocking 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:** $\boldsymbol{Sn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Sn(OH)_2(s)}$ White ppt $\boldsymbol{Sn(OH)_2(s) + 4OH^{-}(aq) \rightarrow Sn(OH)_6^{4-}(aq)}$ 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:** $\boldsymbol{Zn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Zn(OH)_2(s)}$ White ppt $\boldsymbol{Zn(OH)_2(s) + 2OH^{-}(aq) \rightarrow Zn(OH)_4^{2-}(aq)}$ 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 $\boldsymbol{MnO_2}$ *or* $\boldsymbol{Mn_2O_3}$ **Ionic equation:** $\boldsymbol{Mn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Mn(OH)_2(s)}$ White ppt **<u>On standing:</u>** $\boldsymbol{2Mn(OH)_2(s) + O_2(g) \rightarrow 2MnO_2{\cdot}H_2O(s)}$ (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:** $\boldsymbol{Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)}$ White ppt $\boldsymbol{Al(OH)_3(s) + OH^-(aq) \rightarrow Al(OH)_4^-(aq)}$ (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:** $\boldsymbol{Pb^{2+}(aq) + 2OH^-(aq) \rightarrow Pb(OH)_2(s)}$ White ppt $\boldsymbol{Pb(OH)_2(s) + 2OH^-(aq) \rightarrow Pb(OH)_4^{2-}(aq)}$ Plumbate ion (colourless)
Mg^{2+} Ca^{2+}	To a solution of $Mg^{2+}orCa^{2+}$ions in a test-tube add NaOH drop wise until in excess	White precipitate insoluble in excess (conc. Solutions of $Mg^{2+}orCa^{2+}$) **Ionic equation:** $\boldsymbol{Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)}$ White ppt $\boldsymbol{Ca^{2+}(aq) + 2OH^-(aq) \rightarrow Ca(OH)_2(s)}$ 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:** $\boldsymbol{Cu^{2+}(aq) + 2OH^-(aq) \rightarrow Cu(OH)_2(s)}$ A pale blue ppt **N.B** The pale blue ppt turns black on heating due to formation of CuO

		$\boldsymbol{Cu(OH)_2(s) \rightarrow CuO(s) + H_2O(l)}$
Co^{2+}	To a solution ofCo^{2+}ions in a test-tube add NaOH drop wise until in excess	A pale or light blue precipitate insoluble in excess **Ionic equation:** $\boldsymbol{Co^{2+}(aq) + 2OH^-(aq) \rightarrow Co(OH)_2(s)}$ A pale blue ppt **N.B** The pale blue ppt turns grey-pink (or brown) on standing due to oxidation of $\boldsymbol{Co^{2+}}$to $\boldsymbol{Co^{3+}}$
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:** $\boldsymbol{Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)}$ A dirty green ppt **N.B** <u>Oxidation reaction</u> $4\,\boldsymbol{Fe(OH)_2(s)} + O_2(g) \rightarrow Fe_2O_3.2H_2O(s)$
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:** $\boldsymbol{Ni^{2+}(aq) + 2OH^-(aq) \rightarrow Ni(OH)_2(s)}$ A green ppt
Fe^{3+}	To a solution ofFe^{3+}ions in a test-tube add NaOH drop wise until in excess	A brown or yellow precipitate insoluble in excess. **Ionic equation:** $\boldsymbol{Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)}$ 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: $Cr^{3+}(aq) + 3OH^-(aq) \rightarrow Cr(OH)_3(s)$ A grey-green ppt **In excess:** $Cr(OH)_3(s) + 3OH^-(aq) \rightarrow Cr(OH)_6^{3-}(aq)$ Chromite ion **(dark green solution)**
$Cr_2O_7^{2-}$	To a solution of $Cr_2O_7^{2-}$ ions in a test-tube add NaOH drop wise until in excess	The solution changes from orange to yellow $Cr_2O_7^{2-}$ present, $Cr_2O_7^{2-}$ converted to CrO_4^{2-} $Cr_2O_7^{2-}(aq) + 2OH^-(aq) \rightarrow 2CrO_4^{2-}(aq) + H_2O(l)$

SUMMARY

Add sodium hydroxide solution drop-wise until in excess

Observation	Deduction
• No observable change. A colourless gas with a chocking 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	$Pb^{2+}, Al^{3+}, Zn^{2+}, Sn^{2+}, Sn^{4+}$ probably present
• White precipitate insoluble in excess	Ca^{2+}, 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

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:** $\mathbf{Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)}$ White ppt $\mathbf{Zn(OH)_2(s) + 4NH_3(aq) \rightarrow Zn(NH_3)_4^{2+}(aq) + 2OH^-(aq)}$ 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:** $\mathbf{Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)}$ 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:** $\mathbf{Pb^{2+}(aq) + 2OH^-(aq) \rightarrow Pb(OH)_2(s)}$ 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:** $\mathbf{Mg^{2+}(aq) + 2OH^-(aq) \rightarrow Mg(OH)_2(s)}$ White ppt $\mathbf{Ca^{2+}(aq) + 2OH^-(aq) \rightarrow Ca(OH)_2(s)}$ 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:** $\boldsymbol{Sn^{2+}(aq) + 2OH^-(aq) \rightarrow Sn(OH)_2(s)}$ 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 $\boldsymbol{MnO_2}$ *or* $\boldsymbol{Mn_2O_3}$ **Ionic equation:** $\boldsymbol{Mn^{2+}(aq) + 2OH^-(aq) \rightarrow Mn(OH)_2(s)}$ White ppt **<u>On standing:</u>** $\boldsymbol{2Mn(OH)_2(s) + O_2(g) \rightarrow 2MnO_2 \cdot H_2O(s)}$ (brown ppt)
Cu^{2+}	To a solution ofCu^{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:** $\boldsymbol{Cu^{2+}(aq) + 2OH^-(aq) \rightarrow Cu(OH)_2(s)}$ A pale blue ppt $\boldsymbol{Cu(OH)_2(s) + 4NH_3(aq) \rightarrow Cu(NH_3)_4^{2+}(aq) + 2OH^-(aq)}$ 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:** $\boldsymbol{Fe^{2+}(aq) + 2OH^-(aq) \rightarrow Fe(OH)_2(s)}$ A dirty green ppt **<u>N.B</u>** <u>Oxidation reaction</u> $4\,\mathbf{Fe(OH)_2(s)} + O_2(g) \rightarrow Fe_2O_3.2H_2O(s)$
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	$Mg^{2+}Pb^{2+}, Al^{3+}, Ba^{2+}orSn^{2+}$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	$\boldsymbol{Co^{2+}}$present
• A green precipitate soluble in excess forming a pale blue solution	$\boldsymbol{Ni^{2+}}$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 chocking 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 chocking 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 chocking 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* $Cr_2O_7^{2-}$
➢ 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* $Ag(NH_3)_2^+$ *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) ie. OH^-, PO_4^{3-}, $C_2O_4^{2-}$, 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	$Cr_2O_7^{2-}$ *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 chocking 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 flours 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	$Cr_2O_7^{2-}$ *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	*produced.*
✓ Black solid precipitated, colourless pungent gas produced heavily in air. Violet vapour on heating, litmus paper bleached	I^- Present, *HI produced. Purple vapour is iodine (I_2)*
✓ Blue solid turns white	Cu^{2+} present, formation of an hydrous sulphate from a hydrated salt
✓ Red or pink solid turns blue	Co^{2+} present, formation of an hydrous sulphate from hydrated salts
✓ Dark brown solid yields a colourless gas that relights a glowing splint and leaves a white residue on heating	*PbO_2 present. Pb^{4+} converted to* Pb^{2+} ($PbSO_4$), O_2 produced.
✓ Black solid on heating liberates a colourless gas that re-lights a glowing splint. White solid left	*MnO_2 present MnO_2 changed to $MnSO_4$* $MnO_2(s) + 2H_2SO_4(aq) \rightarrow MnSO_4(aq) + O_2(g) + 2H_2O(l)$
✓ Orange-red solid yields a colourless odourless gas that re-lit a glowing splint	*Pb_3O_4 present. Pb_3O_4 reduced to $PbSO_4$*

9. REACTION WITH SODIUM CARBONATE SOLUTION.

TEST: add Na_2CO_3 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 Fe^{2+} 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, $Fe(OH)_3$ pptd. $Fe_2(CO_3)_3$, 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. $CO_3^{2-}+ 2H_2O \longrightarrow H_2CO_3 + 2OH^-$ $NH_4^+ + OH^- \longrightarrow NH_3+H_2O$
• 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	$Cr_2O_7^{2-}$ present.$Cr_2O_7^{2-}$ 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 NH_3(aq)	Cl^- probably present. AgCl pptd. AgCl forms soluble $Ag(NH_3)_2^+$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	Agl pptd I^-Present
❖ Red ppt from yellow solution	CrO_4^{2-} present Ag_2CrO_4pptd
❖ Red ppt from orange solution	$Cr_2O_7^{2-}$ Ag_2CrO_4 pptd.
❖ Black ppt	S^{2-}present, Ag_2S pptd
❖ Sliver mirror(sometimes brownish ppt) observed on warming	Ag pptd, Fe^{2+}probably present i.e. $Fe^{2+} + Ag^+ \rightarrow Fe^{3+} + Ag(s)$.

11. REACTIONS WITH LEAD (II) NITRATE SOLUTION

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

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

o White ppt insoluble on heating; insoluble in dil HNO_3	SO_4^{2-} Present
o 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
o 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
o Yellow ppt, turning orange on heating	CrO_4^{2-} or $Cr_2O_7^{2-}$present, $PbCrO_4$pptd.
o 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 $2Cu^{2+}(aq)+4I^-(aq)\rightarrow Cu_2I_2\ (s) + I_2(aq)$
➢ 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_2pptd Pb^{2+}present,PbI_2pptd $(PbI_4)^{2-}$,solublecomplex formed i.e.$PbI_2(s)+ 2I^-(aq)\rightarrow PbI_4^{2-}(aq)$
➢ Solution turns red/brown	Fe^{3+}, I_2 liberated

NOTE:

i. Some oxidizing agents such as CrO_4^{2-} or $Cr_2O_7^{2-}$,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

ii. 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.

iii. Iodine liberated may be detected by:
- Boiling the solution or suspension , forming purple vapor of I_2
- Adding starch, forming deep blue colour
- Adding CCI_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 (CrO_4^{2-})solution turns green-blue or orange ($Cr_2O_7^{2-}$) solution turns green Note: If I^- present, I_2 is liberated in the presence of H^+, dark brown colour of I_2 will mask the green Cr^{3+}, solid I_2 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 H_2S 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_4Cl/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_4Cl/NH_3)	$Fe^{3+}$from neutral, alkaline or acidified Fe^{3+}ions H_2S oxidized to S and Fe^{3+} reduced to Fe^{2+}
f) A blue solution giving black ppt. buffered solution with NH_4Cl/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_2S oxidized to S and Cr^{6+}reduced to Cr^{3+}

solution)	
h) Orange solution giving muddy green suspension (from neutral test solution)	$Cr_2O_7^{2-}$, 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 $Pb^{2+}(aq) + 2I^-(aq) \rightarrow PbI_2(s)$ A yellow ppt
	• Add dilute hydrochloric acid solution	• A white precipitate which dissolves on warming to give a colourless solution and reforms on cooling $Pb^{2+}(aq) + 2Cl^-(aq) \rightarrow PbCl_2(s)$ 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 $Pb^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow PbCrO_4(s)$ 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:** $Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$ 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:** $Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$ A pale blue ppt $Cu(OH)_2(s) + 4NH_3(aq) \rightarrow Cu(NH_3)_4^{2+}(aq) + 2OH^{-}(aq)$ Tetra ammine copper(II) ions **Deep blue solution**
	• Add potassium or sodium iodide solution	• A white precipitate is formed in a brown solution $2Cu^{2+}(aq) + 4I^{-}(aq) \rightarrow Cu_2I_2(s) + I_2(aq)$
	• Add potassium thiocyanate solution	• Reddish- brown precipitate
	• Add potassium hexacyanoferrate (II) solution ($K_4Fe(CN)_6$)	• A reddish- brown gelatinous ppt of $Cu_2Fe(CN)_6$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:** $Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)$ White ppt $Zn(OH)_2(s) + 2OH^-(aq) \rightarrow Zn(OH)_4^{2-}(aq)$ Zincate ion (colourless)
	• Add ammonia solution drop-wise until in excess	• White precipitate soluble in excess to form a colourless solution **Ionic equation:** $Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)$ A white ppt $Zn(OH)_2(s) + 4NH_3(aq) \rightarrow Zn(NH_3)_4^{2+}(aq) + 2OH^-(aq)$ 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 $Zn(OH)_2$ 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 chocking 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 $Fe.Fe(CN)_6^-$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. $H_2O_2 + 2OH^- + 2Fe^{3+} \rightarrow 2H_2O + 2Fe^{2+} + O_2$

Ca^{2+}	• 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_4solution.	• 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 ofNH_4Cl	• White ppt of $CaCO_3$
Ba^{2+}	• Add potassium chromate solution	• A pale yellow ppt of $BaCrO_4$
	• Flame test: treat a little solution containing Ba^{2+} **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_4solution.	• 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 $CaCO_3$
Al^{3+}	• 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 cm^3 of solution of Al^{3+} **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
Mg^{2+}	• Add a few crystals of NH_4Cl followed by 2-3 drops of disodium hydrogen phosphate (Na_2HPO_4) then finally add $NH_3(aq)$ drop wise till in excess	• White crystalline ppt, insoluble in $NH_3(aq)$.
	• Add a few drops of Magneson followed by little sodium hydroxide solution	• A blue ppt is formed
Ni^{2+}	• Add **ammonia solution** drop wise until in excess	• Pale green ppt (use conc. solution) dissolves in excess giving a pale blue solution of $Ni(NH_3)_4^{2+}$
	• 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 $2Ni^{2+} + Fe(CN)_6^{4-} \rightarrow Ni.Fe(CN)_6$
	• Add potassium hexacyanoferrate(III) solution	• A brown precipitate is formed $3Ni^{2+} + 2Ni(CN)_6^{3-} \rightarrow Ni_3(Fe(CN)_6)_2$

	• Add 2-napthol	• Brown precipitate soluble in dilute hydrochloric acid
	• Add potassium cyanide solution	• A yellowish- green ppt of $Ni(CN)_2$ which dissolves in excess forming a dark yellow solution of $K_2Ni(CN)_4$
Mn^{2+}	• Add a few drops of conc./ dil. HNO_3followed 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 $5BiO_3^- + 2Mn^{2+} + 14H^+ \rightarrow 2MnO_4^- + 5Bi^{3+} + 7H_2O$
	• Add lead (IV) oxide followed by conc. $HNO_3$and boil.	• Colourless solution turns purple $5PbO_2 + 2Mn^{2+} + 4H^+ \rightarrow 2MnO_4^- + 5Pb^{2+} + 2H_2O$
	• Fuse solid containing Mn^{2+} with a large excess fusion mixture(Na_2CO_3/KNO_3)	• Green mass of manganate(VI) $MnO_4^{2-}$is observed
	• Add 1 cm^3 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 $NH_4Cl/NH_3(aq)$ 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, $H_2Co(SCN)$ 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 $Co^{2+} + 2CN^- \rightarrow Co(CN)_2$ $Co(CN)_2 + 4CN^- \rightarrow Co(CN)_4^{2-}$
	• Bubble H_2S gas followed by dil. HCl	• A black ppt insoluble in acid $Co^{2+} + S^{2-} \rightarrow CoS$

	• 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
Cr^{3+}	• Add *NaOH(aq)* drop wise till in excess followed by a few drops of H_2O_2then heat to oxidize the chromite to yellow CrO_4^{2-} then add: • Pentanol(amyl alcohol), then dil. H_2SO_4, shake gently • Lead(II) nitrate to the yellow solution (CrO_4^{2-})	• A green ppt soluble in excess forming a green solution. A yellow solution of CrO_4^{2-} 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 $PbCrO_4$ formed confirms Cr^{3+}
	• Fuse solid with large excess of fusion mixture(Na_2CO_3/ KNO_3)	• Yellow solid of chromate (CrO_4^{2-}) obtained
Sn^{2+}	• Add Na_2S solution	• Brown ppt of SnS
	• *Add potassium manganate(VII) solution*	• Purple color of MnO_4^- turns colourless
	• Add potassium chromate (VI) solution	• Yellow colour of CrO_4^{2-} turns green-blue due to formation of Cr^{3+}
	• Add iron(III) chloride solution	• Yellow- brown, Fe^{3+} solution turns to pale green colour of Fe^{2+}

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
$\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$	• Heat the solid except Na_2C0_3 and K_2C0_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 $\boldsymbol{CO_3^{2-}}$ **and** $\boldsymbol{HCO_3^-}$. 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
$\boldsymbol{SO_4^{2-}}$	• Add dil. *HCl* followed by a few drops of $BaCl_2(aq)$	• White ppt of $BaSO_4$
	• Add dil. HNO_3followed 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 chocking 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 chocking gas (SO_2) produced turns orange $K_2Cr_2O_7$ paper green and damp blue litmus red and bleaches it.
	• Add iodine solution	• The brown color is immediately decolourized
	• Add $FeCl_{3(aq)}$ acidify, warm and add $NaOH_{(aq)}$	• A dark red- brown solution observed becomes almost colourless when hot. Green ppt formed with *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
$S_2O_3^{2-}$	• Add dil. *HCl* and warm	• White (or cream) ppt of sulphur with evolution of bubbles of a colourless gas produced on warming. The colourless chocking gas (SO_2) produced turns orange $K_2Cr_2O_7$ paper green and damp blue litmus red.
	• Add $I_2(aq)$ in *KI* solution	• The brown color of I_2 was immediately decolorized $I_2 + {}_2S_2O_3^{2-} \rightarrow {}_2I^- + S_4O_6^{2-}$
	• Add silver nitrate solution	• Yellow ppt that turns black
	• Add $FeCl_{3(aq)}$ acidify, warm and add $NaOH_{(aq)}$	• Dark purple solution clears when hot, becomes cloudy. Green ppt of $Fe(OH)_2$ with *NaOH(aq)* ${}_2Fe^{3+} + {}_2S_2O_3^{2-} \rightarrow {}_2Fe^{2+} + S_4O_6^{2-}$

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 chocking 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 $FeSO_{4(aq)}$ 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 un affected	• 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 $FeSO_{4(aq)}$followed by dil. *NaOH*	• Mixture turns black, $Fe(H_2O)_5(NO)^{2+}$, 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 $FeSO_{4(aq)}$followed by dil. H_2SO_4	• Dark brown complex ion, $Fe(NO)^{2+}$ formed
$C_2O_4^{2-}$	• Add 2-3 drops of $AgNO_3$ *(aq)* followed by excess NH_3 *(aq).*	• White ppt ($Ag_2C_2O_4$) soluble in excess $NH_3(aq)$, forming a colourless complex $Ag(NH_3)_2^+$

	• 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 70^0C 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
$\boldsymbol{Cr_2O_7^{2-}}$	• 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
$\boldsymbol{S^{2-}}$	• 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 cm^3 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
	• Toa 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 cm^3 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 vopours 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
	• Toa 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 cm^3 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($-NH_2$),for amines
- Others could be; ethers(-O-), alkyl and aryl halides(R-X and Ar-x), esters, amines

PRELIMINARY TESTS

1. **APPEARANCE AT 20^0C (room temperature)**

   - A majority of lower aliphatic compounds such as alcohols, aldehydes, ketones, esters, ethers, amines etc are liquids at 20^0 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^oC

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 an hydrides, 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-dinitrophenylhydrozones 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:

i. 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

ii. 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 1cm^3 of organic substance in a test-tube, add 1or 2 drops of distilled water. Observe, and then add about 3cm^3 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	Alkaline solution, Amine present
• Red litmus turns blue	$RNH_2 + H_2O \leftrightarrow RNH_3^+ + OH^-$ *Salt* of a weak acid and strong base hydrolyzed present $CH_3COONa + H_2O \longleftrightarrow CH_3COOH + NaOH$
• 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 $RNH_3^+ + H_2O \longleftrightarrow H_3O^+ + RNH_2$

• 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 *HCl* 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 un saturated 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.	Ethanedioc (oxalic) acid, or ethanedioate (oxalate) present $(COOH)_2 \longrightarrow CO + CO_2 + H2O$.
➢ H_2 and CO evolved in two stage reaction. H_2 burns if ignited, on strong heating CO is evolved.	Methanoate (formate) present $2H\ COONa \longrightarrow (COONa)_2 + H_2$ $(COONa)_2 \longrightarrow Na_2CO3 + CO$
➢ Ketone evolved (characteristics odour), vapour burns with aluminous flame if ignited	Metal salt of a carboxylic acid $2RCOONa \longrightarrow Na_2\ CO_3 + RCOR$
➢ 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 $1cm^3$ of the substance in a test-tube, add about $5cm^3$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 $RCOOH + OH^- \longrightarrow RCOO^- + H_2O$

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 SODIUMCARBONATE SOLUTION

TEST: To about 0.5g or 1cm^3 of the substance in a test- tube, add about 5cm^3 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. $RNH_3^+ \longleftrightarrow RNH_2 + H^+$

• CO_2 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. $C_6H_5NH_3^+Cl^-$

Note: Na_2CO_3 solution largely behaves like $NaOH$ solution; due to hydrolysis of the CO_3^{2-} *i.e*

$$CO_3^{2-} + H_2O \longleftrightarrow HCO_3^- + OH^-$$

8. REACTION WITH DILUTE HYDROCHLORIC ACID ORSULPHURIC ACID

Test: to a little of the substance in a test tube, add 5cm^{3}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. H_2SO_4 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 cm^3 of the test substance. Add more (1 cm^3) acid, observe carefully in cold the warm gently and observe again. Do not over heat (because chocking white fumes may be observed)

Observation	Deduction
• Effervescence, cold or on gentle warming. Little or no blackening ➢ CO produced (no CO_2), 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 CO_2 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 cm^3 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 addingbromine water untill 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 0.5cm^3 of the test substance add about 4cm^3 of iodine in potassium iodide solution. Then add 2M *NaOH* solution drop wise until the colour of iodine is first discharged, warm and cool.

OBSEVATION	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 $CH_3C{=}O$, $CH_3C{-}OH$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 (chlorofoam), $CHCl_3$ are colourless liquids

Test	Observation	Deduction
• To 1 cm^3 of the test solution add 3 cm^3 of $KI_{(aq)}$,then 10 cm^3 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 0.5gin methanol. To 1 cm^3 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
To1 g or1 $cm^3$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 5cm^3 of the test substance add 1cm^3 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 5cm^3 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 5cm^3 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 i. An aldehyde present ii. 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 1$cm^3$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 cm^3 of an alcohol in a test tube, quickly add 2cm^3 of Lucas reagent. Close the test-tube with a coke 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 5^0C**

TEST; To 0.5g (or 0.5 cm^3) 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 2cm^3 of sodium nitrite solution followed by 2cm^3 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 $R\text{-}NH_2 + HNO_2 \longrightarrow ROH + N_2 + H_2O$
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. $C_6H_5N_2^+ + H_2O \longrightarrow C_6H_5OH + N_2 + H^+$

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-napthol 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