O' LEVEL BASIC QUALITATIVE ANALYSIS EXPOSED

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:

- Colour changes
- Precipitate formed
- Gases evolved
- Sound
- Smell e.t.c

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 O' level chemistry include; $\boldsymbol{Ca^{2+}, Mg^{2+}, Cu^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, NH_4^+, Fe^{2+}, Fe^{3+}}$**.**

ii. **Anions:** These are negatively charged ions. The common anions identified in O' level chemistry include the following; $\boldsymbol{Cl^-, SO_4^{2-}, CO_3^{2-}, NO_3^-, HCO_3^-, SO_3^{2-}}$.

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, $\boldsymbol{Cu^{2+}}$ probably present
ii. Blue	Hydrated salt of copper, $\boldsymbol{Cu^{2+}}$ probably present
iii. Green	Hydrated salts of copper(II) or Iron(II), $\boldsymbol{Cu^{2+}}$or $\boldsymbol{Fe^{2+}}$ probably present
iv. Yellow/brown	Lead(II)oxide(in case of solid) or Iron(III) salt probably present

v.	White solid	Salts of $Ca^{2+}, Mg^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, NH_4^+$, probably present

NB:

- If the substance is a **crystalline solid** then; **Sulphate, Nitrate or a Chloride** is probably present
- If the substance is in **powder** form then **carbonates** are more likely
- If the substance is **deliquescent** then **chloride ion** is probably present

2. 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.

Observation		**Deduction**
i.	Colourless condensate which turns white anhydrous copper(II) sulphate crystals to blue	Water of crystallization(hydrated salt) present or may be $\boldsymbol{HCO_3^-}$ **present**
ii.	Brown fumes of a gas which turn damp blue litmus paper red are evolved	NO_2 gas, NO_3^- probably present
iii.	Coluorless gas with a pungent chocking smell which turns damp red litmus paper blue is evolved and forms white fumes with $HCl\ gas$	Ammonia gas, NH_4^+ probably present
iv.	White sublimate formed on the cooler parts of the test-tube	NH_4^+ salt present
v.	Residue is yellow when hot and white when cold	ZnO formed, Zn^{2+} present
vi.	Residue is brown(Red) when hot and yellow when cold	PbO formed, Pb^{2+} present
vii.	The solid turns from blue/ green to black	CuO is formed, $\boldsymbol{Cu^{2+}}$ **present**
viii.	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
ix.	Colourless gas which relights a glowing splint is given off	Oxygen gas, NO_3^- probably present

x.	Colourless gas which turns damp blue litmus paper red(pink) and lime water milky	CO_2 gas, $\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$ probably present

3. IDENTIFICATION OF GASES

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.

4. 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	$Ca^{2+}, Mg^{2+}, Pb^{2+}, Al^{3+}, Zn^{2+}, NH_4^+$ probably present
ii.	Brown/yellow solid dissolves forming a brown or yellow solution	$\boldsymbol{Fe^{3+}}$ **probably present**
iii.	Green solid dissolves forming a green solution	$\boldsymbol{Fe^{2+}}$ probably present
iv.	Blue/ green solid dissolves giving a blue or green solution	$\boldsymbol{Cu^{2+}}$ probably present

N.B

- All nitrates are soluble in water
- **All** $\boldsymbol{K^+}, \boldsymbol{NH_4^+}, \boldsymbol{Na^+}$ **salts** are soluble in water
- All carbonates are insoluble in water except carbonates of potassium, sodium and ammonium.
- All sulphates are soluble except $CaSO_4$ which is sparingly soluble. $BaSO_4$ and $PbSO_4$ are insoluble in water
- All chlorides are soluble in water except $PbCl_2$ which is sparingly soluble and AgCl which is insoluble in water
- All hydroxides are in soluble except those of sodium, potassium and ammonium. Magnesium and calcium hydroxides are sparingly soluble in water.

5. 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 colourless gas with a pungent chocking smell is given off on warming. The gas turns damp red litmus paper blue and forms dense white fumes with hydrogen chloride gas **Ionic equation:** $\boldsymbol{NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(g) + H_2O(l)}$
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 colourless 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)
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 $\boldsymbol{Al(OH)_3(s) + 2OH^-(aq) \rightarrow Al(OH)_4^-(aq)}$ (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+} **and** 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 **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
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 $\boldsymbol{Cu(OH)_2(s) \rightarrow CuO(s) + H_2O(l)}$
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** oxidation reaction $4\,\mathbf{Fe(OH)_2(s)} + O_2(g) \rightarrow Fe_2O_3.2H_2O(s)$
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:** $\boldsymbol{Fe^{3+}(aq) + 3OH^-(aq) \rightarrow Fe(OH)_3(s)}$ A brown/yellow ppt

SUMMARY

Add sodium hydroxide solution drop-wise until in excess

Observation	Deduction
• No observable change. A colourless gas with a pungent 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, $\boldsymbol{NH_4^+}$ **present**
• White precipitate soluble in excess	$Pb^{2+}, Al^{3+}, Zn^{2+}$ probably present
• White precipitate insoluble in excess	Ca^{2+}, Mg^{2+} probably present
• A pale blue precipitate insoluble in excess	$\boldsymbol{Cu^{2+}}$ **present**
• A dirty green precipitate insoluble in excess and turns brown on standing	Fe^{2+} present
• A brown precipitate insoluble in excess	Fe^{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:** $\boldsymbol{Zn^{2+}(aq) + 2OH^-(aq) \rightarrow Zn(OH)_2(s)}$ White ppt $\boldsymbol{Zn(OH)_2(s) + 4NH_3(aq) \rightarrow Zn(NH_3)_4^{2+}(aq) + 2OH^-(aq)}$ Tetra amine 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:** $\boldsymbol{Al^{3+}(aq) + 3OH^-(aq) \rightarrow Al(OH)_3(s)}$ 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:** $\boldsymbol{Pb^{2+}(aq) + 2OH^{-}(aq) \rightarrow Pb(OH)_2(s)}$ White ppt
Mg^{2+} **and** Ca^{2+}	To a solution of Mg^{2+} or Ca^{2+}ions in a test-tube add ammonia solution drop wise until in excess	White precipitate insoluble in excess **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
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:** $\boldsymbol{Cu^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)}$ A pale blue ppt $\boldsymbol{Cu(OH)_2(s) + 4NH_3(aq) \rightarrow Cu(NH_3)_4^{2+}(aq) + 2OH^{-}(aq)}$ Tetra amine 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 **N.B** oxidation reaction $4\,\mathbf{Fe(OH)_2(s)} + O_2(g) \rightarrow Fe_2O_3.2H_2O(s)$
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:** $\boldsymbol{Fe^{3+}(aq) + 3OH^{-}(aq) \rightarrow Fe(OH)_3(s)}$ A brown/yellow ppt

SUMMARY

Add ammonia solution drop-wise until in excess

Observation	Deduction
• White precipitate soluble in excess	Zn^{2+} present
• White precipitate insoluble in excess	$Ca^{2+}, Mg^{2+} Pb^{2+}, Al^{3+}$ probably present
• A pale blue precipitate soluble in excess to form a deep blue solution	$\boldsymbol{Cu^{2+}}$ **present**
• A dirty green precipitate insoluble in excess and turns brown on standing	Fe^{2+} present
• A brown precipitate insoluble in excess	Fe^{3+} present

6. **CONFIRMATORY TESTS FOR CATIONS.**

CATIONS	CATIONS	OBSERVATION
$\boldsymbol{Pb^{2+}}$	• Heat suspected solid	• Residue is brown when hot and yellow when cold
	• Add potassium iodide solution	• A yellow precipitate $\boldsymbol{Pb^{2+}(aq) + 2I^{-}(aq) \rightarrow PbI_2(s)}$ A yellow ppt
	• Add dilute hydrochloric acid solution	• A white precipitate which dissolves on warming to give a colourless solution and reforms on cooling $\boldsymbol{Pb^{2+}(aq) + 2Cl^{-}(aq) \rightarrow PbCl_2(s)}$ A white ppt
	• Add dilute sulphuric acid solution	• A white ppt of $\boldsymbol{PbSO_4}$ is formed

	• Add potassium chromate solution	• A yellow precipitate of potassium 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 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 amine copper(II) ions **Deep blue solution**
	• Add potassium or sodium iodide solution	• A white precipitate is formed in a brown solution $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow Cu_2I_2(s) + I_2(aq)$
Zn^{2+}	• Heat suspected solid	• Residue is 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	• A 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 amine zinc(II) ions **colourless solution**
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 was evolved
	• Heat the suspected solid	• A white sublimate formed at the cooler parts of the test-tube
Fe^{2+}	• Add potassium hexacyanoferrate(III) solution	• A deep blue ppt is formed
Fe^{3+}	• Add potassium hexacyanoferrate(II) solution	• A deep blue ppt is formed
	• Add potassium or ammonium thiocyanate solution	• Deep red solution is formed
	• Add potassium or ammonium thiocyanate solution	• Deep red solution is formed

7. **DETECTION OF ANIONS**

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

ANION	Test	Observation	Deduction
$\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$	To the solid add dilute acid	Effervescence of a colourless gas which turns lime water milky and damp blue litmus red	Carbon dioxide gas, $\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$ **probably present**
	Heat the solid except Na_2C0_3 and K_2C0_3	Evolution of a colourless gas which turns lime water milky and damp blue litmus red	Carbon dioxide gas, $\boldsymbol{CO_3^{2-}}$ **or** $\boldsymbol{HCO_3^-}$ **probably present**
$\boldsymbol{SO_4^{2-}}$	Add barium nitrate followed by dilute nitric acid	White ppt insoluble in the acid $\boldsymbol{Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)}$	$\boldsymbol{SO_4^{2-}}$ **present**
	Add barium chloride followed by dilute nitric acid	White ppt insoluble in the acid $\boldsymbol{Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)}$	$\boldsymbol{SO_4^{2-}}$ **present**
	Add lead(II) nitrate solution and warm the mixture	White ppt insoluble on warming $\boldsymbol{Pb^{2+}(aq) + SO_4^{2-}(aq) \rightarrow PbSO_4(s)}$	$\boldsymbol{SO_4^{2-}}$ **present**
$\boldsymbol{NO_3^-}$	Heat the suspected salt	Brown fumes of a gas are produced and a colourless gas which relights a glowing splint	Nitrogen dioxide gas and oxygen gas evolved, $\boldsymbol{NO_3^-}$ present
	Add cold freshly prepared Iron(II) sulphate solution followed by concentrated sulphuric acid drop by drop down the side of the test-tube	Brown ring is formed at the interface between the acid and the mixture	$\boldsymbol{NO_3^-}$ present
$\boldsymbol{SO_3^{2-}}$	Add lead(II)nitrate solution followed by dilute nitric acid	White ppt formed soluble in the acid forming a colorless solution	$\boldsymbol{SO_3^{2-}}$**present**

	Add barium nitrate(chloride) followed by few drops of nitric(hydrochloric) acid	White ppt formed soluble in the acid forming a colorless solution	$\boldsymbol{SO_3^{2-}}$ **present**
$\boldsymbol{Cl^-}$	Add silver nitrate solution	White ppt formed $Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$	$\boldsymbol{Cl^-}$present
	Add silver nitrate solution followed by excess ammonia	White ppt formed which dissolves in excess ammonia	$\boldsymbol{Cl^-}$present
	Add lead(II) nitrate solution and warm the mixture	White ppt soluble on warming and insoluble on cooling $Pb^{2+}(aq) + 2Cl^-(aq) \rightarrow PbCl_2(s)$	$\boldsymbol{Cl^-}$present

8. **DIFFERENTIATING SOME ANIONS**

i. $\boldsymbol{CO_3^{2-}}$ **and** $\boldsymbol{HCO_3^-}$

Reagent: Magnesium sulphate solution

Observation: With CO_3^{2-}, a white precipitate of $MgSO_4$ is formed while with HCO_3^- there is no observable change

ii. $\boldsymbol{SO_4^{2-}}$ **and** $\boldsymbol{Cl^-}$

Reagent: lead (II) nitrate solution followed by warming

Observation: With SO_4^{2-}, a white precipitate insoluble on warming is formed while with Cl^- a white precipitate soluble on warming is formed

iii. $\boldsymbol{SO_3^{2-}}$**and** $\boldsymbol{SO_4^{2-}}$

Reagent: lead (II) nitrate solution followed by dilute nitric acid

Observation: With SO_4^{2-}, a white precipitate insoluble in the acid is formed while with $\boldsymbol{SO_3^{2-}}$ a white precipitate soluble in the acid is formed forming a colourless solution