



# Qualitative Analysis

The practical book for:

**General Chemistry (I) course**

**For 1<sup>st</sup> year University Students.**

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# **Qualitative Analysis**

**A practical book**

## INTRODUCTION

Chemical analysis can be either qualitative or quantitative in character. In qualitative analysis we want to know elements or characteristic chemical species are present. Although it might be possible to gain some idea of how much of each component is present. The major point of the qualitative analysis is simply to determine whether the component of interest is present at a level above the minimum required for detection. On the other hand, in a quantitative analysis we are interested in the relative amounts of the components present.

This book is about qualitative and quantitative inorganic analysis. It contains the background theory as well as experimental procedures that the student will need to carry out a laboratory program of chemical analysis.

Cations and their analyses are discussed group by group and the same on dealing with anions. A systematic concise procedure for detection of each group anions and cations is summarized in a specific table followed by tables prepared for student practice for each cation or anion.

A simple systematic part for quantitative analysis is also included in this book.

Some important analyses are described for student. By doing so this book slows student to observe descriptive chemical reacts and to test principles spelled out in the pages of their textbooks. In

addition, students are challenged to apply their knowledge in the laboratory.

### ABBREVIATIONS OF COMMON USE

Word or sentence	Abbreviation	Word or sentence	Abbreviation
Ammonium	Amm.	Equivalent weight	Eq.wt.
Bolling point	S.P.	Example	ex.
Cubic	c.c.	And so on	etc.
centimeter	Cm.	Gram	gm.
Concentrated	Conc.	That is to say	i.e.
Concentration	Concn.	Insoluble	Insol.
Compound	cpd.	Molecular weight	Mol.wt.
Conical flask	C.F.	Melting point	M.P.
Dilute	dil.	Potassium	Pot.
Different	diff.	Solution	Soln.
Precipitate	ppt.	Sodium	Sod.
to precipitate	pptate.	Test tube	T.T.
precipitation	pptn.	Volume	Vol.
Saturates	Sat.	Excess	xss.
Soluble	sol.	Round Bottom Flask	R.B.F
Heat	$\Delta$	Flat Bottom Flask	F.B.F

## CHEMICAL ANALYSIS

Chemical analysis is divided into two main classes:

### I) Qualitative analysis:

This type of analysis involves the investigation and identification of substances in its simplest or complicated forms. In this connection, the constituent element or group of elements, and the way in which these elements are combined to form the substance, are identified.

### II) Quantitative Analysis:

The object of quantitative analysis is to determine the actual amounts of the constituents of a compound, and the amount of material dissolved in solution.

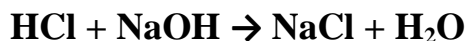
Depending upon the tools used, or the procedures followed to perform the analysis, it can be classified into 3 main classes:

- A. **Volumetric analysis**, i.e. determination of the constituents by titration.
- B. **Gravimetric analysis**: i.e. determination of the constituents by precipitation.
- C. **Instrumental analysis**: i.e. determination of the constituents by the use of instruments and apparatus.

## QUALITATIVE ANALYSIS

### Radicals:

When an acid e.g. HCl is made to react with a base, e.g NaOH. a salt, NaCl and water are formed according to the following equation:



acid + base  $\rightarrow$  salt + water

The part of the salt, which is derived from the base, Na, is called the "basic radical", whereas the other part, which is derived from the acid, is termed "acidic radical".

### Analysis of a simple salt:

Dealing with a simple salt, the various steps enquired for its identification can be gathered as follows:

1. Preliminary tests.
2. Analysis for acidic radicals.
3. Analysis for basic radicals.

#### 1) preliminary Tests:

These tests are usually termed "Dry Tests" since they are carried out on the material in its dry state. These tests cannot be considered as final and conclusive, but however they furnish valuable indication of the presence of certain acid and basic radicals.

The preliminary tests include:

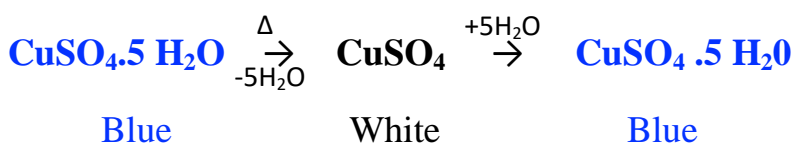
**A. Physical properties:**

This involves the examination of the physical properties of the salt such as color, crystalline form and solubility of the substance is checked with the following solvents in the same order as cited:

1. Water; cold then hot.
2. Dil. HCl; cold then hot.
3. Conc. HCl; cold then hot.
4. Dil. HNO<sub>3</sub>; cold then hot.
5. Conc. HNO<sub>3</sub>; cold then hot.
6. Aqua Regie: 3 vols. conc. HCl + 1 vol. conc. HNO<sub>3</sub>
7. Effect of Heat:

When a small amount of the solid is heated in a hard dry test tube, some changes may occur which can throw some light on the identity of the salt. These changes may include:

1. Decomposition e.g. copper carbonate  
$$\text{CuCO}_3 \xrightarrow{\Delta} \text{CuO (black)} + \text{CO}_2$$
2. Change of color, e.g. Zinc oxide is colored white, but when heated it becomes yellow and upon cooling it returns white again.
3. Loss of water of crystallization, e.g. Copper sulphate



4. **Sublimation:** Some compounds e.g ammonium chloride transforms directly from the solid state to the vapor state.

### **B. Flame Test**

It was found that some metals give characteristic colors to the flame when their volatile compound e.g. chloride are heated on a platinum wire in the oxidizing zone of the flame.

**In this experiment:** A clean Pt wire (or glass rode) is dipped into a conc. HCl then in the dry substance and heated in the flame. Different colors are produced by different basic radicals:

Copper: Green                      Calcium: Brick red  
 Strontium: Crimson red          Barium: Apple green  
 Sodium: Golden yellow          Potassium: Pale violet.

### **ACIDIC RADICALS**

Our study is restricted here to only fourteen acidic radicals of the most famous ones. They can be classified towards: dil. HCl and hot conc. H<sub>2</sub>SO<sub>4</sub> as follows:

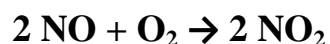
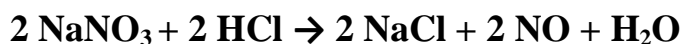
<b>I- Dil. HCl group</b>	<b>II- Hot conc. H<sub>2</sub>SO<sub>4</sub> group</b>	<b>III- Miscellaneous group</b>
Carbonate CO <sub>3</sub> <sup>-2</sup>	Chloride Cl <sup>-</sup>	Sulphate SO <sub>4</sub> <sup>-2</sup>
Blicarbonate HCO <sub>3</sub> <sup>-</sup>	Bromides Br <sup>-</sup>	Borate B <sub>4</sub> O <sub>7</sub> <sup>-2</sup>
Sulphide S <sup>-2</sup>	Iodides I <sup>-</sup>	Arsenate AsO <sub>4</sub> <sup>-3</sup>
Sulphite SO <sub>3</sub> <sup>-2</sup>	Nitrates NO <sub>3</sub> <sup>-</sup>	Phosphate PO <sub>4</sub> <sup>-3</sup>
Thiosulphate S <sub>2</sub> O <sub>3</sub> <sup>-2</sup>		
Nitrites NO <sub>2</sub> <sup>-</sup>		



It is to be noted that all radicals of one group reacts with the so called " Group Reagent " which is dil. HCl in the first group and hot conc. H<sub>2</sub>SO<sub>4</sub> in the second group to give off characteristic gases and vapors. The radicals in the miscellaneous group do not react with either dil. HCl or conc. H<sub>2</sub>SO<sub>4</sub>

### **1. Dilute Hydrochloric acid Group**

This group includes radicals of unstable acids which upon reaction with dil. HCl decompose readily giving off characteristic gases as illustrated by the equations:



Experiment	Carbonate Na <sub>2</sub> CO <sub>3</sub>	Biocarbonate e.g. NaHCO <sub>3</sub>	Sulphide e.g Na <sub>2</sub> S	Sulphite e.g. Na <sub>2</sub> SO <sub>3</sub>	Thiosulphate e.g. Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	Nitrite NO <sub>2</sub> <sup>-</sup>
solid + dil. HCl heat if necessary	Effervescence & evolution of CO <sub>2</sub> which detected by rendering lime water turbid	As carbonate	Evolution of H <sub>2</sub> S gas detected by its bad odor and blackening a lead acetate paper	Evolution of SO <sub>2</sub> gas detected by its suffocating odor & rendering an acid chromate paper green	As sulphite but soln. becomes turbid due to sepn. of colloidal Sulphur	Red-brown fumes due to combination of NO and O <sub>2</sub> of air & turns blue
2) Soln. + AgNO <sub>3</sub>	white ppt of Ag <sub>2</sub> CO <sub>3</sub> sol. in dil. HNO <sub>3</sub>	As carbonate but on heating	Black ppt. of Ag <sub>2</sub> S sol. in hot dil. HNO <sub>3</sub>	White ppt. of Ag <sub>2</sub> SO <sub>3</sub> sol. in xss. of sulphite and in NH <sub>4</sub> OH	White ppt. Ag <sub>2</sub> SO <sub>3</sub> sol. in xss. AgNO <sub>3</sub> . It turns yellow, orange then black Ag <sub>2</sub> S	White ppt. from conc. solns.
3) Soln. + BaCl <sub>2</sub> soln.	White ppt. of BaCO <sub>3</sub> sol. in dil. HCl and HNO <sub>3</sub>	As carbonate but on heating		White ppt. of BaSO <sub>3</sub> sol. in dil. HCl and HNO <sub>3</sub>	White ppt. from conc. solns.	

Experiment	Observation & results
<p><b>i- Carbonates &amp; Biocarbonate:</b></p> <p>1) Soln.+ HgCl<sub>2</sub> soln.</p> <p>2) Soln. + HgSO<sub>4</sub> soln.</p> <p><b>ii-Sulphides:</b></p> <p>1) Soln. + NaOH soln. + sodium nitroprusside Na<sub>2</sub>Fe(CN)<sub>5</sub>NO</p> <p>2) Soln. + lead acetate Pb(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub></p> <p><b>iii- Sulphites. Thiosulphates and Nitrites:</b> (Reducing properties)</p> <p>1) One drop of KMO<sub>4</sub> + dil H<sub>2</sub>SO<sub>4</sub> soln.</p> <p>2) One drop of K<sub>2</sub>Cr<sub>2</sub>O<sub>2</sub> + dil H<sub>2</sub>SO<sub>4</sub> soln.</p> <p>3) One drop of I<sub>2</sub> + dil H<sub>2</sub>SO<sub>4</sub> soln.</p> <p><b>iv-Nitrites: (Oxidizing properties)</b></p> <p>1) Ferrous sulphate (FeSO<sub>4</sub>) soln. + dil.H<sub>2</sub>SO<sub>4</sub> soln.</p> <p>2)1ml. pot. Thiocyanate (KCNS)+1ml dil. H<sub>2</sub>SO<sub>4</sub> soln.</p> <p><b>v- Sulphites and Thiosulphates</b> Soln. + Ferric chloride Soln. (FeCl<sub>3</sub>)</p> <p>1) Thiosulphate</p> <p>2) Sulphite</p>	<p>Reddish brawn ppt. of basic mercuric carbonate HgCO<sub>3</sub></p> <p>Purple coloration due to form of a comp. structured as: Na<sub>4</sub>Fe (CN)<sub>5</sub>NO.</p> <p>Black ppt. sol. in hot dil. NHO<sub>3</sub></p> <p>Decolorization due to form. Of MnSO<sub>4</sub>. Development of green color due to Cr<sup>+3</sup> decolorization due to form of iodide</p> <p>Brown color due to formation of Fe(NO)SO. Blood red color which discharges on boiling.</p> <p>Dark violet color disappears on warming or standing</p> <p>Red color suffering no changes on standing.</p>

### Detection of Carbonate $\text{CO}_3^{-2}$

Experiment	Observation	Result
Solid + dil. HCl		
Soln. + $\text{AgNO}_3$ soln.		
Soln.+ $\text{BaCl}_2$ soln.		
Soln.+ $\text{HgCl}_2$ soln.		
Soln. + $\text{HgSO}_4$ soln.		
Physical Prop.		

### Detection of the Sulphide $S^{2-}$

Experiment	Observation	Result
Solid + dil. HCl		
Soln. + $AgNO_3$ soln.		
Soln. + NaOH soln.		
Sod. Nitophrouside		
Soln. + lead acetate		
Physical Prop:		

### Detection of the Sulphite $\text{SO}_3^{-2}$

Experiment	Observation	Result
Solid + dil. HCl		
Soln.+ AgNO <sub>3</sub> soln.		
Soln. + BaCl <sub>2</sub> soln.		
Soln. + drop of KMnO <sub>4</sub> + dil. H <sub>2</sub> SO <sub>4</sub>		
Soln. + drop of K <sub>2</sub> Cr <sub>2</sub> O <sub>2</sub> Soln. + dil. H <sub>2</sub> SO <sub>4</sub>		
Soln. + drop of I <sub>2</sub>		
Soln. + dil. H <sub>2</sub> SO <sub>4</sub>		
Physical Prop. :		

### Detection of the Thiosulphate $S_2O_3^{-2}$

Experiment	Observation	Result
Solid + dil. HCl (Heat gently)		
Soln.+ AgNO <sub>3</sub> soln.		
Soln. + drop of KMnO <sub>4</sub> soln. + dil. H <sub>2</sub> SO <sub>4</sub>		
Soln.+ drop of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> soln. + dil. H <sub>2</sub> SO <sub>4</sub>		
Soln. + FeSO <sub>4</sub> soln. + dil. H <sub>2</sub> SO <sub>4</sub>		
Soln. + 1ml. KCNS soln. +1 ml. dil. H <sub>2</sub> SO <sub>4</sub>		
Physical Prop. :		

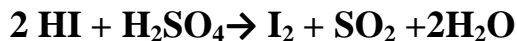
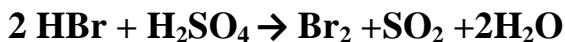
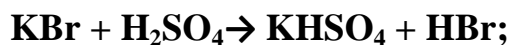
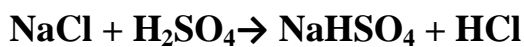
### Detection of the nitrite radical $\text{NO}_2^-$

Experiment	Observation	Result
Solid + dil. HCl		
Soln.+ $\text{AgNO}_3$ soln.		
Soln.+ $\text{BaCl}_2$ soln.		
Soln. + drop of $\text{KMnO}_4$ + dil. $\text{H}_2\text{SO}_4$		
Soln.+ drop of $\text{K}_2\text{Cr}_2\text{O}_7$ soln. + dil. $\text{H}_2\text{SO}_4$		
Soln.+ drop of $\text{I}_2$ soln. + dil. $\text{H}_2\text{SO}_4$		
Physical Prop. :		



## **II-Concentrated Sulphuric Acid Group:**

The group includes radicals belonging to acids such stronger than those of the previous group, and therefore they are not affected by dil. HCl. When the salt is treated with conc. H<sub>2</sub>SO<sub>4</sub>, the corresponding acids are liberated which may further decompose as following:



**(A) General Tests for Hot Conc. H<sub>2</sub>SO<sub>4</sub> group.**

<b>Experiment</b>	<b>Chloride e.g. NaCl</b>	<b>Bromide e.g. NaBr</b>	<b>Iodide e.g. KI</b>	<b>Nitrate e.g. NaNO<sub>3</sub></b>
Solid + conc. H <sub>2</sub> SO <sub>4</sub> heat	Evolution of HCl gas detected by rendering blue litmus paper red	Evolution of red brown vapors of Br <sub>2</sub> and HBr and soln. becomes orange	Evolution of vapors of I <sub>2</sub> detected by turning starch paper blue.	Red brown vap. of NO <sub>2</sub> after long heating
2 Solid + H <sub>2</sub> O <sub>2</sub> + conc. H <sub>2</sub> SO <sub>4</sub> heat	Evolution of Cl <sub>2</sub> gas detected by its yellow green color and bleaching action on litmus paper	Evolution of Br <sub>2</sub> gas detected by its red brown color and bleaching action on litmus and turning starch paper yellow	Same as above	
3 Soln.+ AgNO <sub>3</sub> soln.	White curdy ppt. sol. in NH <sub>4</sub> OH and insol. in dil.	Pale yellow curdy ppt. sol. in conc. NH <sub>4</sub> OH and insol. in dil. HNO <sub>3</sub>	Yellow curdy ppt. insol. in both NH <sub>4</sub> OH and NaOH	
Soln., lead acetate soln.	White ppt. of PbCl <sub>2</sub> sol. in boiling water	White cryst. ppt. in boiling water	Yellow ppt. of PbI <sub>2</sub> sol. in boiling water	

**(B) Special Tests for Hot Conc. H<sub>2</sub>SO<sub>4</sub> group.**

Experiment	Observation & Results
<p><b>(i) Bromides &amp; Iodides:</b></p> <p>Soln. Chlorine water</p> <p><b>Bromide</b> -----</p> <p><b>Iodide</b> -----</p> <p>Reactn. mix + few mls. chloroform (CHCl<sub>3</sub>)</p> <p><b>Bromide</b>-----</p> <p><b>Iodide</b>-----</p> <p><b>(ii) Iodides</b></p> <p>1-Soln.+ HgCl<sub>2</sub> soln.</p> <p>2-Soln. + CuSO<sub>4</sub> soln.</p> <p>3. Reactn. mix. + Na<sub>2</sub>SO<sub>3</sub> soln.</p> <p><b>(iii) Nitrates:</b></p> <p><b>1-Brown Ring Test:</b></p> <p>soln. + cold sat. FeSO<sub>4</sub> soln. + 2 ml. Conc. H<sub>2</sub>SO<sub>4</sub>, drop by drop to form a separate layer.</p> <p><b>2- Ammonia Test</b></p> <p>soln. + Zinc dust (Al) + NaOH, Heat.</p>	<p>Solution becomes pale orange.</p> <p>Solution becomes yellow brown</p> <p>Chloroform layer is colored red brown.</p> <p>Chloroform layer is colored violet.</p> <p>A scarlet red ppt. of HgI<sub>2</sub> sol. in xss. soln.</p> <p>A ppt. of cuprous iodide Cu<sub>2</sub>I<sub>2</sub> colored dirty brown due to liberation of I<sub>2</sub></p> <p>Disappearance of brown color of iodine.</p> <p>A brown ring is formed at the interface due to the formation of the compound Fe(NO)SO<sub>4</sub></p> <p>Ammonia gas (NH<sub>3</sub>) evolves which is detected by its odor and by turning a red litmus paper blue</p>

### Detection of the Chloride Radical (Cl<sup>-</sup>)

Experiment	Observation	Result
Solid + conc. H <sub>2</sub> SO <sub>4</sub> heat.		
Solid+MnO <sub>2</sub> +conc. H <sub>2</sub> SO <sub>4</sub> heat.		
Soln. +AgNO <sub>3</sub> soln.		
Soln.+ lead acetate soln.		
Physical Prop.:		

### Detection of the Bromide Radical ( $\text{Br}^-$ )

Experiment	Observation	Result
Solid +conc. $\text{H}_2\text{SO}_4$ heat.		
Solid + $\text{MnO}_2$ + conc. $\text{H}_2\text{SO}_4$ heat.		
Soln. + $\text{AgNO}_3$ soln.		
Soln.+ lead acetate soln.		
Soln. + chlorine water.		
reaction mixture of 5 + few ml's of chloroform.		
Physical Prop.:		

## Detection of the Iodide Radical (I<sup>-</sup>)

Experiment	Observation	Result
1. Solid + conc. H <sub>2</sub> SO <sub>4</sub> , heat.		
2. Solid + MnO <sub>2</sub> + conc. H <sub>2</sub> SO <sub>4</sub> heat.		
3. Soln. + AgNO <sub>3</sub> soln.		
4. Soln.+ lead acetate soln.		
5. Soln. + chlorine water.		
6. Reaction mixture of 5 + few ml's of chloroform.		
7. Soln. +HgCl <sub>2</sub> soln.		
8. Soln. CusO <sub>4</sub> soln.		
9. Reaction mixture of 8 + Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> soln.		
<b>Physical Prop.:</b>		

### Detection of the Nitrate Radical ( $\text{NO}_3^-$ )

Experiment	Observation	Result
1. Solid + conc. $\text{H}_2\text{SO}_4$ heat.		
2. Soln. + $\text{AgNO}_3$ Soln.		
3. Brown ring test: Soln. + saturated $\text{FeSO}_4$ soln. + 2 ml. conc. $\text{H}_2\text{SO}_4$ drop by drop without shaking.		
4. Ammonia test: Soln. + Zn(or Al) + NaOH heat.		
Physical Prop.:		

### III Miscellaneous Group

This group includes the remaining acid radicals which are not affected by either dil. HCl or hot conc. H<sub>2</sub>SO<sub>4</sub>

#### (A) General Tests:

Experiment	Sluphate e.g. Na <sub>2</sub> SO <sub>4</sub>	Borate e.g. Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	Phosphate e.g. Na <sub>2</sub> HPO <sub>4</sub>
1. Soln. + AgNO <sub>3</sub> soln.	Sluphate e.g. Na <sub>2</sub> SO <sub>4</sub>	White ppt. AgBO <sub>3</sub> sol. in NH <sub>4</sub> OH and acetic acid	Yellow ppt. of Ag <sub>3</sub> PO <sub>4</sub> sol. in NH <sub>4</sub> OH and dil. HNO <sub>3</sub>
2. Soln. + BaCl <sub>2</sub>	White ppt. of BaSO <sub>4</sub> from Conc. solns. only	White ppt. of Ba <sub>3</sub> (BO <sub>3</sub> ) <sub>2</sub> from conc. solns. sol. in xss. BaCl <sub>2</sub> and dil. HCl and HNO <sub>3</sub>	White ppt. of BaHPO <sub>4</sub> From neutral soln. sol. in dil. mineral acids and acetic acid.
3. Soln.+Magnesia mixture (MgCl <sub>2</sub> +NH <sub>4</sub> Cl+H <sub>4</sub> OH)			White ppt. of HgNH <sub>4</sub> PO <sub>4</sub> sol. in acetic acid and in mineral acids



### B ) Special Tests:

Experiment	Observation & Results
i- Sulphate: Soln. + acetate soln.	White ppt. of $\text{PbSO}_4$ sol. In Amm. acetate.
ii- Borate: 1) Soln. + one drop of ph.ph.	Pink color which is decolorized by dil. glycerol. The color appears on heating and disappears on cooling.
2) Conc. soln. conc. HCl.	White ppt. of boric acid $\text{H}_3\text{BO}_4$
iii- Phosphate: 1) Soln. + amm. molybdate + 2 ml. conc. $\text{HNO}_3$	Cream yellow ppt. sol. in dil. HCl. But insol. in acetic acid
2) Soln. $\text{FeCl}_3$ soln.	

### Detection of the Sulphate Radical ( $\text{SO}_4^{-2}$ )

Experiment	Observation	Result
1. Soln. + $\text{AgNO}_3$ soln.		
2. Soln.+ $\text{BaCl}_2$ soln.		
3. Soln. + lead acetate soln.		
4. Soln + $\text{SrCl}_2$ soln.		
Physical Prop.:		

### Detection of the Borate Radical ( $B_4O_7^{--}$ )

Experiment	Observation	Result
1. Soln. + $AgNO_3$ soln.  2. Soln. + One drop of ph.ph. and heating.  3. Conc. soln. + conc. HCl.  Physical Prop. :		

### Detection of the Phosphate Radical ( $\text{HPO}_4^{2-}$ )

Experiment	Observation	Result
1. Soln. + $\text{AgNO}_3$ soln.		
2. Soln. + Magnesia mix. ( $\text{Mg} + \text{NH}_3\text{Cl} + \text{NH}_4\text{OH}$ ).		
3. Soln. + amm. Molybdate+ 2 ml. conc. $\text{HNO}_3$ .		
4. Soln. + $\text{FeCl}_3$ soln.		
Physical Prop.:		

General Scheme for identification of the Acid Radical carry on the following experiments in the given order, to define the group to which the radical belongs:

### 1-Test for the presence of dil. HCl group:

Solid + dil. HCl, heat if necessary, characteristic of gases:

Observation	Conclusion	Confirm tests
a) Effervescence & evolution of a gas that causes turbidity for lime water.	Gas is CO <sub>2</sub> & radical may be carbonate or bicarbonate.	(1) If the salt is insol. So the radical is carbonate without any confirmation. (2) +ve result on cold: <b>carbonate</b> . (3) +ve result after heating: <b>bicarbonate</b> . * <b>BaCl<sub>2</sub></b> ---White ppt. sol. in dil. HCl. * <b>HgCl<sub>2</sub></b> ----Red brown ppt. * <b>AgNO<sub>3</sub></b> ----White ppt. sol. in dil. HCl
b) A gas of bad odor & blackens a lead acetate paper.	Gas is H <sub>2</sub> S & radical may be sulphide	<b>AgNO<sub>3</sub></b> → Black ppt. <b>HaOH</b> + sod. nitroprusside -- violet color. <b>CdSO<sub>4</sub></b> --- canary yellow ppt.
c) A gas of suffocating odor and turns acid dichromate paper green.	Gas is SO <sub>2</sub> & radical may be sulphite	* <b>AgNO<sub>3</sub></b> -----White ppt. <b>Acid I<sub>2</sub></b> -----Decolor <b>BaCl<sub>2</sub></b> ----- White ppt. sol. in dil. HCl
d) A gas as in (c) but soln. becomes turbid (white yellow).	Gas is SO <sub>2</sub> & turbidity is S & the radical may be thiosulphate.	<b>AgNO<sub>3</sub></b> ----- White ppt. ----- yellow ---- black. <b>I<sub>2</sub></b> -----Decolor. <b>FeCl<sub>3</sub></b> -----Violet color, vanishes gradually
e) A gas that becomes red brown at mouth of tube & soln. becomes blue.	Gas is NO and the radical may be nitrite.	* <b>AgNO<sub>3</sub></b> - White pp. <b>Acid I<sub>2</sub></b> ---Decolor. <b>FeSO<sub>4</sub> + acid</b> ---- Brown color.

## 2-Test for the presence of hot conc. H<sub>2</sub>SO<sub>4</sub> group

Solid + conc. H<sub>2</sub>SO<sub>4</sub>, heat mildly then strongly characteristic of gases.

Observation	Conclusion	Confirm tests
a) Evolution of a gas that turns blue litmus paper into red.	Gas is HCl & the radical may be Cl <sup>-</sup>	*AgNO <sub>3</sub> ----White cruddy ppt. *Pb(Ac) <sub>2</sub> ----- White ppt. sol. in boiling water. *Hg(NO <sub>2</sub> ) <sub>2</sub> ---White silky ppt.
b) Evolution of red brown fumes & soln. becomes red color	Gases are Br <sub>2</sub> & the radical may be bromide	*AgNO <sub>3</sub> ----Pale yellow ppt. *Pb(Ac) <sub>2</sub> ----- White ppt. sol. in boiling H <sub>2</sub> O *MnO <sub>4</sub> <sup>-</sup> + conc. H <sub>2</sub> SO <sub>4</sub> ----- red brown fumes.
c) Evolution of violet fumes which turns starch paper to blue color	Gas are I <sub>2</sub> & The radical may be iodide.	*AgNO <sub>3</sub> -----yellow ppt. *HgCl <sub>2</sub> -----Scarlet red ppt. sol. in the xss iodide *CuSO <sub>4</sub> -----Dirty white ppt. clarifies with S <sub>2</sub> O <sub>3</sub> <sup>-2</sup>
d) Evolution of red brown fumes after long heating.	Gases are NO <sub>2</sub> & the radical may be nitrate	*Brown ring test + ve. *Zn(NaOH)heat ----NH <sub>3</sub> gas. *Cu +conc. H <sub>2</sub> SO <sub>4</sub> -----Red Brown fumes

### 3-Tests for the pressure of the miscellaneous group:

(A) Soln. +  $\text{AgNO}_3 \rightarrow \text{ppt.}$

(B) Soln. +  $\text{BaCl}_2 \rightarrow \text{ppt.}$

Conclusion
<b>May be Sulphate:</b>
<ul style="list-style-type: none"> <li>* (A) Soln. + <math>\text{AgNO}_3 \rightarrow \text{White ppt.}</math></li> <li>* Soln. + <math>(\text{Pb}(\text{Ac})_2</math>-- White ppt from conc. soln.</li> <li>* (B) Soln. + <math>\text{BaCl}_2 \rightarrow \text{White ppt. insol. in HCl}</math></li> </ul>
<b>May be Borate:</b>
<ul style="list-style-type: none"> <li>* (A) Soln. + <math>\text{AgNO}_3 \rightarrow \text{White ppt. sol. in NH}_4\text{OH \&amp; acetic Acid}</math></li> <li>* Soln. + <math>\text{HgCl}_2</math>----- Red brown ppt.</li> <li>* (B) Soln. + <math>\text{BaCl}_2 \rightarrow \text{White ppt. sol. in xss. BaCl}_2 \&amp; \text{HCl}</math></li> <li>* Soln. + Conc. HCl -----White crystals.</li> </ul>
<b>May be Phosphate:</b>
<ul style="list-style-type: none"> <li>* (A) Soln. + <math>\text{AgNO}_3 \rightarrow \text{Yellow ppt. sol. in NH}_4\text{OH \&amp; Acids.}</math> *</li> <li>* Soln. + <math>\text{FeCl}_3</math> -- Cream yellow ppt.</li> <li>* (B) Soln. + <math>\text{BaCl}_2 \rightarrow \text{White ppt. sol in acids.}</math></li> <li>* Soln. + Mg mix-----White ppt</li> <li>* Soln. + Amm. molybdate + conc.<math>\text{HNO}_3</math> --- Yellow ppt.</li> </ul>

Unknown No. (    )

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		



## **BASIC RADICALS**

The famous basic radicals can be classified into six groups according to their behavior towards certain reagent. The "Group reagent" which will precipitate down any one of the members of a given group when present in solution. The following table includes this classification.

It is to be noted that most of the tests for basic radicals are carried out in solution. If the substance is insoluble in water, its solubility will be tried with the previously mentioned solvents in the given order.

The use of these solvents changes only the acid radical and has no effect on the basic radical or the salt under test.

### Analytical Group

Group	Reagent	Metals	Cations
I	dil. HCl	Sliver (Hg)Mercurous Lead	Ag <sup>+</sup> Hg <sub>2</sub> <sup>+2</sup> Pb <sup>++</sup>
II	H <sub>2</sub> S (Acidic medium) HCl	(Hg) Mercuric Copper Bismuth Cadmium	Hg <sup>+2</sup> Cu <sup>++</sup> BI <sup>+++</sup> Cd <sup>+2</sup>
III	[NH <sub>4</sub> Cl + NH <sub>4</sub> OH]	Iron Aluminum Chromium	Fe <sup>+2</sup> , Fe <sup>+3</sup> Al <sup>+3</sup> Cr <sup>+3</sup>
IV	(NH <sub>4</sub> ) <sub>2</sub> S + [NH <sub>4</sub> Cl+NH <sub>4</sub> OH]	Zinc Manganese Nickel Cobalt	Zn <sup>+2</sup> Mn <sup>+2</sup> Ni <sup>+2</sup> Co <sup>+2</sup>
V	(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> + [NH <sub>4</sub> Cl+NH <sub>4</sub> OH]	Calcium Barium Strontium	Ca <sup>+2</sup> Ba <sup>+2</sup> Sr <sup>+2</sup>
VI	No specific reagent	Magnesium Potassium Sodium Ammonium	Mg <sup>+2</sup> K <sup>+</sup> Na <sup>+</sup> NH <sub>4</sub> <sup>+</sup>

**(dil. HCl group)**

It includes basic radicals for silver ( $\text{Ag}^+$ ), Mercurous ( $\text{Hg}_2^{2+}$ ), and lead ( $\text{Pb}^{++}$ ). The group reagent is dil. HCl which forms ppt. of the corresponding chlorides.

<b>Tests</b>	<b>Silver e.g. <math>\text{AgNO}_3</math></b>	<b>Lead e.g <math>\text{Pb(NO}_3)_2</math></b>	<b>Mercurous e.g. <math>\text{Hg}_2(\text{NO}_3)_2</math></b>
1) Soln. + dil HCl acid.	White curdy ppt. of $\text{AgCl}$ sol. in $\text{NH}_4\text{OH}$ and $\text{Na}_2\text{S}_2\text{O}_3$ but insol. in dil. $\text{HNO}_3$	White ppt. of $\text{PbCl}_2$ from cold conc. solns. sol. in hot water and separates on cooling.	White ppt. of $\text{Hg}_2\text{Cl}_2$ insol. in cold dil. acids but sol. in aqua Regia ( $3\text{HCl}:1\text{HNO}_3$ ) conc.
2) Soln. + $\text{H}_2\text{S}$ soln.	Black ppt. of $\text{Ag}_2\text{S}$ insol. in $\text{HN}_4\text{OH}$ but sol. in $\text{HNO}_3$	Black ppt. of $\text{PbS}$ sol. in hot dil. $\text{HNO}_3$	Immediate black ppt. of $\text{Hg}_2\text{S}$ .
3) Soln. + KI soln.	Yellow ppt. of $\text{AgI}$ insol. in both $\text{NH}_4\text{OH}$ & dil. $\text{HNO}_3$	Yellow ppt. of $\text{PbI}_2$ sol. in boiling water and separates on cooling in golden yellow plates.	Yellowish green ppt. of $\text{Hg}_2\text{I}_2$ sol. in xss KI.
4) Soln + $\text{K}_2\text{CrO}_4$ soln.	Brown-red ppt. of $\text{Ag}_2\text{CrO}_4$ sol. in dil. $\text{HNO}_3$ & $\text{HN}_4\text{OH}$ but insol. in dil. acetic acid	Yellow ppt. of $\text{PbCrO}_4$ insol. in acetic acid but sol. in Hydroxides & $\text{HNO}_3$	Brown ppt. of $\text{Hg}_2\text{CrO}_4$ on cold which becomes red on boiling.
5) Soln. + NaOH soln.	Brown ppt. of $\text{Ag}_2\text{O}$ insol. in xss. NaOH but sol. in xss. $\text{NH}_4\text{OH}$	White ppt. of $\text{Pb(OH)}_2$ sol. in xss. NaOH	Black ppt. of $\text{Hg}_2\text{O}$ insol. in xss NaOH

**(B) Special tests for dil. HCl Group**

<b>Experiment</b>	<b>Observation and results</b>
i-Silver: 1) Soln. +2 drops $\text{Na}_2\text{S}_2\text{O}_3$	White ppt. sol. in xss. $\text{Na}_2\text{S}_2\text{O}_3$ , it turns yellow, orange then black.
2) Soln. + sod. phosphate	White ppt. of $\text{AgOH}$ sol. in Xss $\text{KOH}$ to Form $\text{K Ag (OH)}_2$ .
ii-Lead: soln. + dil. $\text{H}_2\text{SO}_4$	White ppt. of $\text{PbSO}_4$
iii- Mercurous mercury:	
1) Soln. + pot. Nitrate soln.	Yellow ppt. sol. in $\text{HNO}_3$ and $\text{NH}_4\text{OH}$
2)Soln.+stannous chloride soln.	Dark grey ppt. of metallic Hg

### Detection of Silver $\text{Ag}^+$

Experiment	Observation	Result
1) Soln. + dil. HCl		
2) Soln.+ $\text{H}_2\text{S}$ soln.		
3) Soln. + KI soln.		
4)Soln.+ $\text{K}_2\text{CrO}_4$ soln.		
5) Soln.+ NaOH soln.		
6) Soln. + 2 drops of $\text{Na}_2\text{S}_2\text{O}_3$ soln.		
7) Soln. + sod. phosphate soln.		
Physical Prop.:		

### Detection of Lead ( $\text{Pb}^{++}$ )

Experiment	Observation	Result
1) Soln. + dil. HCl		
2) Soln. + $\text{H}_2\text{S}$ soln.		
3) Soln. + KI soln.		
4) Soln. + $\text{K}_2\text{CrO}_4$ soln.		
5) Soln. + NaOH soln.		
6) Soln. + dil. $\text{H}_2\text{SO}_4$		
Physical Prop.:		

### Detection of Mercurous ( $\text{Hg}_2^{++}$ )

Experiment	Observation	Result
1) Soln.+ dil. HCl		
2) Soln. + $\text{H}_2\text{S}$ soln.		
3) Soln. + KI soln.		
4)Soln.+ $\text{K}_2\text{CrO}_4$ soln.		
5)Soln. + NaOH soln.		
6) Soln.+ $\text{KNO}_3$ soln.		
7) Soln. + stannous chloride soln.		
Physical Prop.:		

## **Group II (H<sub>2</sub>S Group)**

The group includes metal cations which are precipitated as Sulphides by H<sub>2</sub>S hot HCl-acidified solution. The members of the group are classified into two subgroups according to the solubility of their sulphides in hot yellow ammonium sulphide as follows:-

**1) Subgroup (II A) or Copper Group:** It contains mercuric mercury, bismuth, copper and cadmium whose sulphides are insol. in hot yellow amm. sulphide.

**2) Subgroup (II B) or Arsenics Group:** It contains arsenic antimony and tin, the sulphides of which are soluble in hot yellow amm. sulphide, Experimental details are omitted.

### **Group (II A) Copper Group**

The reaction of the members of the copper group with the group reagent which consists of dil. HCl and H<sub>2</sub>S to form insol. Sulphides is represented by the following equations:-





**(A) General Tests for gr. II A**

<b>Tests</b>	<b>Mercuric e.g. HgCl<sub>2</sub></b>	<b>Bismuth e.g. Bi(NO<sub>3</sub>)<sub>3</sub></b>	<b>Copper e.g. CuSO<sub>4</sub></b>	<b>Cadmium e.g. CdSO<sub>4</sub></b>
1) Soln. + HCl dil. (till Acidic) Warm + H <sub>2</sub> S	White ppt. at first of Hg <sub>3</sub> Cl <sub>2</sub> S <sub>2</sub> which changes to yellow brown and finally black (HgS)	Brown ppt. of Bi <sub>2</sub> S <sub>3</sub> insol. In yellow amm. Sulphide but sol. in hot dil. HNO <sub>3</sub>	Black ppt. of CuS insol. in yellow amm. sulphide but sol. in dil. HNO <sub>3</sub> + KCN soln.	Yellow ppt. of CdS insol. in yellow amm. sulphide but sol. in hot dil. HNO <sub>3</sub>
2) Soln. + NaOH soln.	Red-brown ppt. of the basic chloride that changes to yellow HgO	White ppt. of Bi(OH) <sub>3</sub> sol. in dill mineral acids	Blue ppt. of Cu(OH) <sub>2</sub> changes to black ppt. of CuO on heating	White ppt. of Cd(OH) <sub>2</sub>
3) Soln. + NH <sub>4</sub> OH soln.	White ppt. of amino mercuric chloride to yellow HgO	White basic slat insol. in xss. NH <sub>4</sub> OH	A pale blue ppt. of the basic salt sol. in xss. NH <sub>4</sub> OH to give a deep blue color.	White pp. of Cd(OH) <sub>2</sub> sol. in xss. NH <sub>4</sub> OH
4) Soln. + KI soln.	Scarlet red ppt. of HgI <sub>2</sub> sol. in xss. KI to form K <sub>2</sub> HgI <sub>4</sub>	Dark brown ppt. of BiI <sub>3</sub> sol. in xss. KI to give a yellow soln. upon dilution, the brown ppt. reform.	White ppt. of Cu <sub>2</sub> I <sub>2</sub> together with a brown solution due to liberation of I <sub>2</sub>	

### (B) Special Tests for gr.( IIA)

Experiment	Results and observations
<p>i- Mercuric Hg: 1-Soln. + Sn Cl<sub>2</sub> soln. 2-Soln. + (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> soln.</p>	<p>White ppt. of HgCl<sub>2</sub> which changes to grey-black ppt. with xss. of SnCl<sub>2</sub> Red-brown ppt. of basic carbonate.</p>
<p>ii- Bismuth: 1-Soln. + xss. water  2-Soln.+ sod. Stannite Na<sub>2</sub>(SnO<sub>2</sub>) (Stannous Chloride +xss. NaOH)</p>	<p>A white ppt. of the basic salt Bi(OH)<sub>3</sub>, sol. in dil mineral acids, but insol. in tartaric acid soln. (contrary to Sb). and in solutions of alkali hydroxides (contrary to Sb). Black ppt. of finely divided Bi.</p>
<p>iii-Copper: 1-Soln. + pot. Ferrocyanide K<sub>2</sub>Fe(CN)<sub>6</sub>  2-Soln.+ KCN</p>	<p>Red brown ppt. of cupric ferrocyanide Cu<sub>2</sub>Fe(CN)<sub>6</sub> insol. in dil acids but sol. in as NH<sub>4</sub>OH to a blue soln. yellow ppt. of Cu(CN)<sub>2</sub> which decomposes to white Cu<sub>2</sub>(CN)<sub>2</sub> that dissolves. In xss. KCN to form K<sub>3</sub>Cu(CN)<sub>4</sub>.</p>
<p>iv-Cadmium 1-Soln. +KCN  2-Soln. + K<sub>2</sub>Fe(CN)<sub>6</sub></p>	<p>white ppt. of Cd(CN) + sol. in xss. KCN, on passing H<sub>2</sub>S in the resulting solution, a yellow ppt. of CdS forms (contrary to Cu). White ppt. of cadmium ferrocyanide.</p>

**Unknown No. ( )**

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		

## **GROUP III**

### **Ammonium Hydroxide Group**

Members of this group are Ferrous Iron, Ferric Iron, Chromium and Aluminum, The group reagent is composite consists of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  that form insoluble hydroxides as illustrated by the following equations:



**(A) General Tests for Gr. III**

<b>Tests</b>	<b>Ferrous e.g. FeSO<sub>4</sub></b>	<b>Ferric e.g. FeCl<sub>3</sub></b>	<b>Aluminum e.g. Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub></b>	<b>Chromium e.g. CrCl<sub>3</sub></b>
1) Soln. + NH <sub>4</sub> Cl + NH <sub>4</sub> OH till alkaline	Dirty green ppt. of Fe(OH) <sub>2</sub> insol. in alk. Hydroxides but sol. in dil. mineral acids	Red-brown gelatinous ppt. of Fe(OH) <sub>3</sub> insol. in alk. Hydroxides but disol. in acids.	White gelatinous ppt. of Al(OH) <sub>3</sub> slightly sol. in xss. NH <sub>4</sub> OH	Grey green gelat. ppt. of Cr(OH) <sub>3</sub> slightly sol. in xss. NH <sub>4</sub> OH to give a violet soln. & reppt. on boiling.
2) Soln. + NaOH	Same as above	Same as above	White gelate. ppt. Al(OH) <sub>3</sub> sol. in xss. NaOH to Sod. aluminate NaAlO <sub>2</sub>	Grey green ppt. sol. in acids & in xss. NaOH to give green soln. of NaCrO <sub>2</sub>
3) Soln + NH <sub>2</sub> HPO <sub>4</sub>		Pale yellow ppt. FePO <sub>4</sub> insol. in acetic acid but sol. in mineral acid & alk. solns.	White gelate. ppt. of AlPO <sub>4</sub> insol. in acetic but sol. in mineral acids + alk. solns.	Green ppt. of CrPO <sub>4</sub> sol. in dil. mineral acids & insol. in acetic acid.

**(B) Special Tests for Gr. III**

<b>Experiment</b>	<b>Results and observations</b>
<p><b>1- Ferrous Iron</b></p> <p>1) Soln. + pot. Ferricyanide <math>K_3Fe(CN)_6</math>.</p> <p>2) soln. + KCN -----</p> <p>3) soln. + <math>H_2S</math> + <math>NH_4 OH</math>-----</p> <p>4) soln. + pot. Ferrocyanide <math>K_4[Fe(CN)_6]</math>. -----</p> <p>5) soln. + pot.thiocyanate KCNS</p> <p><b>ii- Ferric iron</b></p> <p>1) soln. + <math>K_3Fe(CN)_6</math>-----</p> <p>2) soln. + KCNS -----</p> <p>3) soln. + (neutral) + <math>Na_2S_2O_3</math></p> <p>4) soln. + sod. acetate.</p> <p><b>iii- Aluminium:</b></p> <p>1) soln. + <math>Na_2CO_3</math></p> <p>2) soln. (neutral) + xss. sod. acetate then boil.-----</p> <p><b>iv- Chromium</b></p> <p>soln. + xss. NaOH + small quantity of sod. peroxide.</p>	<p>Deep blue ppt. (Turnbul's blue).</p> <p>Yellowish white ppt. of ferrous cyanide sol. in xss. KCN to form a yellow soln. of <math>K_4Fe(CN)_6</math>.</p> <p>A black ppt. of FeS.</p> <p>NO ppt.</p> <p>NO color.</p> <p>An intense blue ppt. of Prussian blue.</p> <p>Blood red colour decolorized by <math>HgCl_2</math></p> <p>A violet, red colour that quickly disappears.</p> <p>White ppt. of <math>Al(OH)_3</math> sol. in xss. of <math>Na_2CO_3</math></p> <p>A voluminous ppt. of basic acetate <math>Al(Ac)_2</math>.</p> <p>A yellow solution.</p>

**Unknown No. ( )**

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		

**Unknown No. ( )**

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		



**Unknown No. ( )**

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		

**Unknown No. ( )**

<b>Experiment</b>	<b>Observation</b>	<b>Result</b>
Physical Prop. :		

## **GROUP(IV)**

### **Ammonium Sulphide Group**

This group contains divalent metal cations of Zinc, Manganese, Nickel and Cobalt. the group reagent consists of  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{S}$  or  $\text{H}_2\text{S}$  that forms insol. sulphide in alk. medium according to the following equations: -



### (A) General Tests for Gr. IV

Tests	Zinc	Manganese	Cobalt	Nickel
1) Soln. + NH <sub>4</sub> Cl + NH <sub>4</sub> OH (till just alk.) + H <sub>2</sub> S	White ppt. of ZnS sol. in dil. mineral acids.	Fleshy ppt. of MnS, becomes brown in air.	Black ppt. of CdS. sol. in conc. HNO <sub>3</sub> with separation of S.	Black ppt. of NiS sol. in hot conc. HNO <sub>3</sub>
2) Soln. +NaOH or NH <sub>4</sub> OH.	White gelat. ppt. of Zn(OH) <sub>2</sub> sol. in xss. alk. forming Na <sub>2</sub> ZnO <sub>2</sub> and also sol. in dil. acids.	White ppt. of Mn(OH) <sub>2</sub> . becomes brown in air insol. in xss. alkail	A blue ppt. is formed on cold, whereas a pink ppt. which turns brown is formed with xss. alkali and heating.	Green ppt. of Ni(OH) <sub>2</sub> insol. in NaOH but sol.in NH <sub>4</sub> OH and amm. slats to form green- blue solns.
3) Soln. + Na <sub>2</sub> CO <sub>3</sub> Or (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	White ppt. of basic carbonate sol. in xss. (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	White ppt. of MnCO <sub>3</sub>	Red ppt. of basic salt sol. in xss. (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	Apple green ppt. NiCO <sub>3</sub> sol. in xss. (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>
4) slon. + KCN	White ppt. of Zn(CN) <sub>2</sub> sol. in xss. KCN	Brown ppt. of Mn(CN) <sub>2</sub> sol. in xss. KCN to a brown soln. that gives green ppt. on heating.	Red-brown ppt. of Co(CN) <sub>2</sub> sol. in xss. KCN to a brown soln. of K <sub>4</sub> Co(CN) <sub>6</sub>	Light green ppt. Ni(CN) <sub>2</sub> sol. In xss. KCN to give K <sub>2</sub> Ni(CN) <sub>4</sub>

## (B) Special Tests for Gr. IV

Experiment	Results and observations
<b>i- Zinc</b>	
1) Soln. + NH <sub>4</sub> Cl sod. phosphate (Na <sub>2</sub> HPO <sub>4</sub> )	White ppt. of Zn(NH <sub>4</sub> )PO <sub>4</sub> sol. in NH <sub>4</sub> OH and dil. acids.
2) soln. + pot. ferrocyanide	White ppt. of Zn <sub>2</sub> Fe(CN) <sub>6</sub>
<b>ii- Manganese</b>	
1) soln. + NH <sub>4</sub> OH + ferrocyanide.	Fleshy ppt. of Mn(NH <sub>4</sub> )PO <sub>4</sub>
2) soln. + pot. Ferrocynide.	Brown ppt. of Mn <sub>2</sub> Fe(CN) <sub>6</sub>
3) soln + lead peroxide + conc. HNO <sub>3</sub> , boil, dil with H <sub>2</sub> O, allow to stand and settle.	The supernatant liquid is colored violet due to formation of MnO <sub>4</sub> <sup>-</sup>
<b>iii- Cobalt:</b>	
1) soln. + NH <sub>4</sub> CNS	Blue solution due to form of (NH <sub>4</sub> ) <sub>2</sub> Co(CNS) <sub>4</sub> .
2) (soln. + (acetic) + xss. KNO <sub>2</sub>	Yellow ppt. of K <sub>3</sub> Co(NO <sub>2</sub> ) <sub>6</sub>
3) soln. + pot. ferrocyanide	Brown-red ppt. of Co <sub>3</sub> Fe(CN) <sub>6</sub> insol. in dil. HCl
<b>iv- Nickel</b>	
1) soln. + NH <sub>4</sub> OH till (alk.) + dimethyl glyxime (C <sub>4</sub> H <sub>8</sub> O <sub>2</sub> H <sub>2</sub> )	Red ppt. of Nickel dimethyl glyoxime

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

**Unknown No. ( )**

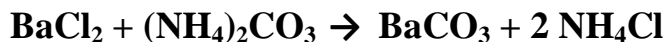
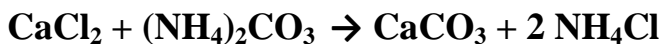
Experiment	Observation	Result
Physical Prop. :		



## GROUP V

### Ammonium Carbonate Group

This group presents the only example where metals of the same group of periodic table form an independent group of basic radicals. It contains the divalent metal cations of calcium, strontium and barium which occupy group II A in the periodic table. The group reagent consists of  $\text{NH}_4\text{Cl}$  (avoid xss.), then  $\text{NH}_4\text{OH}$  till just alk., and  $(\text{NH}_4)_2\text{CO}_3$  which form insol. Carbonates from hot ( $60^\circ \text{C}$ ) solutions.



### (A) General Tests for Gr. V

Tests	Calcium	Strontium	Barium
1) Soln. + NH <sub>4</sub> Cl (few) + NH <sub>4</sub> OH (till just alk.) + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> .	White ppt. of CaCO <sub>3</sub> sol. in dil. acids.	White ppt. of SrCO <sub>3</sub> sol. in dil. acids.	White ppt. of BaCO <sub>3</sub> sol. in dil. acid
2) Soln. + Ca(OH) <sub>2</sub> just alk.) + Amm. oxalate.	White ppt. of CaC <sub>2</sub> O <sub>4</sub> insol. in acetic & sol. in mineral acids.	White ppt. of SrC <sub>2</sub> O <sub>4</sub> insol. in acetic & sol. in mineral acid.	White ppt. of BaC <sub>2</sub> O <sub>4</sub> sol. in mineral acids.
3) Soln. + CaSO <sub>4</sub> soln.	No ppt.	White ppt. of SrSO <sub>4</sub> insol. in (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	Heavy white ppt. of BaSO <sub>4</sub> insol. in dil. acids.
4) soln. + sod. carbonate	White ppt. sol. in mineral acids & acetic acid.	Same as Ca.	Same as Ca
5) soln. (conc.) + K <sub>2</sub> CrO <sub>4</sub>	Yellow ppt. of CaCrO <sub>4</sub> sol. in acetic acid.	Yellow ppt. sol. in acetic acid.	Yellow ppt. from neutral soln. insol. in acetic acid
Flame test	Brick---red color.	Crimson red color	Apple green color

### (B) Special Tests for Group V

	Experiment	Observation & Result
<b>Calcium:</b> Soln. (conc.)	+ NH <sub>4</sub> Cl + pot. Ferrocyanate	A white ppt. is formed especially on standing

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

## GROUP VI

### The Alkali Group

This group contains those cations which are not pptd. by any of the previously mentioned group reagents. Those radicals are sodium ( $\text{Na}^+$ ), potassium ( $\text{K}^+$ ), ammonium ( $\text{NH}_4^+$ ) and Magnesium ( $\text{Mg}^{++}$ ).

Experiment	Observation + Results
<b>i-Magnesium (Mg):</b> 1) soln. + $\text{NH}_4\text{Cl}$ + $\text{NH}_4\text{OH}$ + $\text{Na}_2\text{HPO}_4$ . 2) soln. + $\text{NaOH}$ 3) soln. + $(\text{NH}_3)_2\text{CO}_3$ or $\text{Na}_2\text{CO}_3$ .	White ppt. of $\text{Hg}(\text{NH}_4)\text{PO}_4$ sol. in acids.  <b>White ppt. insol in xss. <math>\text{NaOH}</math>, sol. in amm. salts.</b>  White ppt. of heating sol. in amm. salts.
<b>ii- Ammonium (<math>\text{NH}_4^+</math>):</b> 1) solid t conc. $\text{NaOH}$ , heat. 2) soln. + Nessler's reagent $\text{K}_2\text{HgI}_4$ 3) soln. + sat. sod. Hydrogen tartarate (or tartaric acid soln.) 4) soln. + sod. Cobalti nitrite $\text{Na}_3[\text{Co}(\text{NO}_2)_6]$ . 5) soln. + few drops $\text{KI}$ + one drop sat. sod. hypochlorite ( $\text{NaOCl}$ ). 6) Solid, heat.	Ammonia gas evolves detected by, odor and turning red litmus blue. <b>Brown ppt. or brown-yellow coloration.</b>  White ppt. of amm. acid tartarate.  <b>Yellow ppt. of <math>(\text{NH}_4)_3\text{Co}(\text{NO}_2)_6</math>.</b>  A black ppt. of $\text{NaHI}_2$ , is formed  <b>Decompa., evolution of <math>\text{NH}_3</math> gas and condensation of white ppt. on colder part of T.T.</b>

(B) Special Tests for Group (VI)

The Alkali Group

(continued )

Experiment	Results and observations
<b>iii- Potassium (K<sup>+</sup>):</b> 1) soln. + NH <sub>3</sub> Co (NO <sub>2</sub> ) <sub>6</sub>  2) soln. + sat. tartaric acid, <b>scratch.</b>  3) soln. + perchloric acid or NaClO <sub>4</sub> .  4) Carnot's test : one drop dil. <b>Bismuth nitrated + 2 drops dil. Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> + 10 ml alcohol + neutral soln.</b>  5) Flame test.	Yellow ppt. especially on warming.  <b>White ppt. from conc. solns.</b>  White crystalline ppt. of KClO <sub>4</sub> .  <b>A yellow ppt. of pot. Bismuth Thiosulphate.</b>  Pale violet color
<b>iv- Sodium:</b> 1) soln. + Na <sub>3</sub> Co(HO <sub>2</sub> ) <sub>6</sub>  2) soln. + tartaric acid  3) soln.+ pot. dihydrogen antimonite.  4) flame test.	No ppt.  <b>No ppt.</b>  White crystalline ppt. of sod. dihydrogen antimonite after scratching  <b>Golden yellow coloration.</b>

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		



**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

**Unknown No. ( )**

Experiment	Observation	Result
Physical Prop. :		

## Some Important Remarks:

### i-Dissolution:

- 1) Powder the solid and then try its solubility in the solvent scheme previously mentioned in the given order.
- 2) If the solid dissolves in dil. HCl then group I is absent.
- 3) If the substance dissolved in conc. HCl the soln. should be diluted with dil. HCl before adding the group reagent otherwise BiOCl.
- 4) If all reagents up to aqua-regia fail to dissolve the substance. The solid is fused some of its weight with dry  $\text{Na}_2\text{CO}_3$  and extract the fused mass with a little boiling water and filter. Dissolve residue in dil.  $\text{HNO}_3$  and test the resulting soln. for basic radical. The filtrate on the other hand can be sued for the detection of acid radicals.
- 5) A substance may dissolve on heating but reppts. on cooling as  $\text{PbCl}_2$ , so one must cool the solution before carrying out the tests. If any ppt. is formed on cooling, another solvent must bestride.
- 6) If a concentrated acid is used the solution must be diluted with water then cooled.
- 7) It is preferable in case of  $\text{HNO}_3$  & aqua-regia to evaporate till dryness then dissolve residue in water (nitrates interfere with the identification of gr. II, by oxidizing  $\text{H}_2\text{S}$  into yellow-white colloidal S).

## **ii-Some difficulties**

- 1) If there is a bismuth salt, a white ppt. may be formed on adding HCl and thus gr. I may be suspected. Once the ppt. dissolves in xss. water gr. is absent. Better add H<sub>2</sub>S on the white ppt. if it does not turn black then gr. I is absent.
- 2) If a thiosulphate is present a white ppt. is formed sometime after adding HCl which soon turns yellow especially on warming due to separation of colloidal Sulphur which can be separated by boiling the soln. with a piece of filter paper and filtering.
- 3) If a white ppt. is formed upon addition of dil. HCl as a reagent for gr. I, it will be either from gr. I, Bi from Gr. II ppt. disappears it is Bi or Sb. Then add H<sub>2</sub>S, if it turns black it is from gr. (I) otherwise it is Bi or Sb.
- 4) If the concn. of dil. HCl used in gr. It is somewhat higher Cd may escape precipitation with the gr. reagent in this case neutralization of the acidic soln. with NH<sub>4</sub>OH causes appearance of the yellow ppt.
- 5) If the amount of NH<sub>4</sub>Cl added in the reagent of gr III is not sufficient, members of the coming groups may precipitate. In this case add more NH<sub>4</sub>Cl, if ppt. dissolves gr. III is absent; if not gr. III is present.
- 6) Solution must be quite alk. for identification of both gr. (IV) & (V).

7) Bicarbonates may give the carbonate tests on acid due to impurities of carbonates.

8) Testing for nitrates requires prolonged heating with conc.  $\text{H}_2\text{SO}_4$  and adding a piece of Cu metal before ascertaining absence of nitrate.

9) Testing for miscellaneous group requires relatively concentrated solutions.

## GENERAL SCHEME FOR IDENTIFICATION OF SIMPLE INORGANIC UNKNOWNNS

A-Report on the physical properties as:

- 1) Color      2) Crystalline state      3) Solubility (Action on litmus).

B-Identification of the Basic Radical:

Cary on the following ten tests in the given order

1-test for the presence of  $\text{NH}_4^+$  Solid, NaOH soln. conc. heat

Observation	Conclusion	Confirm tests
Evolution of gases that turns red litmus blue and have charact. odor	Gas in $\text{NH}_3$ & the basic radical may be ammonia.	*Nessler's reagent. yellow color or ppt. * $\text{Na}_3[\text{Co}(\text{NO}_2)_6]$ -- yellow Ppt. * $\text{KI} + \text{NaOCl}$ ----- Black ppt.

## 2-Test for the presence of group (I)

**soln. (cold) dil. HCl-----Heavy white ppt.**

Observation	Conclusion	Confirm tests
a- white ppt. sol. in $\text{NH}_4\text{OH}$ & turn violet by heat.	ppt. is $\text{AgCl}$ and the radical may be $\text{Ag}^+$	* $\text{H}_2\text{S}$ ---- black ppt. sol. in $\text{NHO}_3$ and insol. in $\text{NH}_4\text{OH}$ . * $\text{KCN}$ ---- white ppt. sol. in xss. $\text{KCN}$ . * $\text{K}_2\text{CrO}_4$ (neutral)- red brown ppt.
b-white ppt. blackens with	ppt. is $\text{Hg}_2\text{Cl}_2$ and the radical may be $\text{Hg}_2^{+2}$	* $\text{H}_2\text{S}$ --- black ppt. insol. In $\text{NHO}_3$ * $\text{SnCl}_2$ ---- -white ppt. turns grey with xss.
c-white ppt. insol. in $\text{NH}_4\text{OH}$	ppt. is $\text{PbCl}_2$ and the radical may be $\text{Pb}^{++}$	$\text{H}_2\text{S}$ ----- black ppt. sol. in hot $\text{HNO}_3$ $\text{K}_2\text{CrO}_4$ ----- yellow ppt. insol. in acetic and sol. in nitric $\text{NaOH}$ ----- white ppt. sol. in XSS

### 3-Test for group (IIA)

Soln.+ dil. HCl, hot to 60° C + H<sub>2</sub>S -- Colored ppt. insol. in yellow (NH<sub>4</sub>)<sub>2</sub>S

Observation	Conclusion	Confirm tests on soln.
a) Black ppt. insol. in conc. HNO <sub>3</sub>	ppt. is HgS and radical may be Hg <sup>++</sup>	*NaOH-- yellow or red ppt. *KI-----violet ppt. sol. in xss. KI *Na <sub>2</sub> CO <sub>3</sub> -----red brown ppt.
b) Black ppt. sol. in conc. HNO <sub>3</sub> to give a blue soln.	ppt. is CuS and radical may be Cu <sup>++</sup>	*KI----dirty white ppt. *HN <sub>4</sub> OH-----pale blue ppt. sol. in xss. *NH <sub>4</sub> OH + K <sub>4</sub> Fe(CN) <sub>6</sub> ---red brown ppt. insol. in acids and sol. in NH <sub>4</sub> OH
c) Brown ppt. sol in conc. HNO <sub>3</sub> colorless soln.	ppt. is Bi <sub>2</sub> S <sub>3</sub> & radical may be Bi <sup>+3</sup>	*NaOH-- white ppt. that change to yellow on boiling. *H <sub>2</sub> O---- formn. of white ppt. Na <sub>2</sub> SnO <sub>2</sub> -- -- black ppt.
d) Canarian yellow ppt. sol. in conc. HNO <sub>3</sub> to give a colorless soln.	ppt. is CdS & radical may be Cd <sup>++</sup>	NaOH white ppt. insol in xss. KCN- white ppt. sol. in xss. K <sub>3</sub> Fe(CN) <sub>6</sub> -- -white ppt.



**4-Test for group (III) 1 ml. soln. + 2 ml. NH<sub>4</sub>Cl +NH<sub>4</sub>OH (till just alk.) ppt.**

Observation	Conclusion	Confirm tests
a) white gelat. ppt. sol. in NaOH	ppt. is Al(OH) <sub>3</sub> + radical may be aluminum.	*Na <sub>2</sub> CO <sub>3</sub> ---- white ppt. sol. in xss.
b) Dirty green ppt sol. in acids only	ppt. is Fe(OH) <sub>2</sub> and radical may be ferrous	NaCO <sub>3</sub> *Na <sub>2</sub> HPO <sub>4</sub> --- white gelat. pp. sol. in acids & bases.
c) Dirty green ppt. sol. in acids and bases	ppt. is Cr(OH) <sub>3</sub> & radical may be chromium	*(NH <sub>4</sub> ) <sub>2</sub> S ---- white ppt. *pot. Ferricyanide -- Blue ppt KCN----- yellow white ppt. sol. in xss. KCN
d) Red brown ppt.	ppt. is Fe(OH) <sub>3</sub> & radical may be Fe <sup>+3</sup>	KCNS---- No blood red coloration. *NaOH---- Grey green ppt. sol. in xss. H <sub>2</sub> HPO ----green ppt. sol. In dil. Mineral acids. *xss. NaOH+ Na <sub>2</sub> O <sub>2</sub> ----yellow soln. Pot. Ferrocyanide ----- Blue ppt. KCNS -----Blood red colour. Na <sub>2</sub> HO <sub>4</sub> --- cream yellow ppt. insol. in acetic.

**5- Test for group (IV) 1 ml. soln. +1 ml. NH<sub>4</sub>OH (till alk.) + H<sub>4</sub>S (xss.) ppt.**

Observation	Conclusion	Confirm tests
a) white gelat. ppt. sol. in acids.	ppt. is ZnS & radical may be Zinc.	*NaOH---- white ppt. sol. in *KCH----white ppt. sol. in XSS. *NHCl + Na <sub>2</sub> HPO <sub>4</sub> --- white ppt.
b) Fleshy ppt. sol. in acids only.	ppt. is MnS and radical may be Manganese.	*NaOH---white ppt. which changes to brown.. *KCN----brown ppt. sol. in XSS to brown soln.. *Pot. Ferrocyanide--- brown ppt.
c) Black ppt. insol.in acids soln. + dimethyl glyoxime + NH <sub>4</sub> OH red ppt.	ppt. is NiS & radical may be Ni	* NaOH-- Apple green ppt. sol. in acid * KCN--- pale. green ppt. sol. in X *(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> ----- apple green ppt. sol. in xss.
d) Black ppt. insol. in Acids. soln. + NH <sub>4</sub> OH+ dimethyl Glyoxime no ppt.	ppt. is CoS & radical may be Cobalt	*KCNS -- Blue soln. *Acetic acid + xss. KNO <sub>3</sub> ---- yellow ppt. *Acetic acid + xss. KNO <sub>2</sub> ----yellow ppt. *KCN---- pale green ppt. sol. in xss. KCN

**6- Test for group (V) 1 ml. soln. + 1 ml. NH<sub>4</sub>Cl + NH<sub>4</sub>OH( till alk.) + (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> white ppt.**

Observation	Conclusion	Confirm tests
a) white ppt. & brick red color for flame.	ppt. is CaCO <sub>3</sub> & radical may be Calcium.	*CaSO <sub>4</sub> --- -no ppt. *K <sub>2</sub> CrO <sub>4</sub> --yellow ppt. sol. in dil acetic K <sub>3</sub> Fe(CN) <sub>6</sub> (NH <sub>4</sub> Cl)--- white ppt. after time.
b) white ppt. & crimson red color for flame.	ppt. lin SrCO <sub>3</sub> & radical may be Strontium.	*CaSO <sub>4</sub> ---- white ppt. form conc. solutions K <sub>2</sub> CrO <sub>4</sub> ---- yellow ppt. sol. in hot acetic * sod. phosphate -white ppt. sol. in acids.
c) white ppt. & apple green color for flame.	ppt. is Ba CO <sub>3</sub> & radical may be Barium.	*CaSO <sub>4</sub> --- Immediate white ppt. *K <sub>2</sub> CrO <sub>4</sub> -- -- yellow ppt. insol. In acetic *Sod. phosphate--white ppt. sol. in acids.

### 7-Test for group (VI)

$\text{Mg}^{++}$  soln. +  $\text{NH}_4\text{Cl}$  +  $\text{NH}_4\text{OH}$  (alk.)  $\text{Na}_2\text{HPO}_4$  white ppt.

Conclusion	Confirm tests
ppt. is $\text{Mg}(\text{NH}_4)\text{PO}_4$ + radical may be Mg.	* $\text{NaOH}$ ----- white ppt. insol. in xss. $\text{NaOH}$ sol. in amm. salts * $\text{Na}_2\text{CO}_3$ --- white ppt., on heating

### 8-Test for $\text{K}^+$

Carnot's test: One drop dil.  $\text{Bi}(\text{NO}_3)_3$  + 2 drops  $\text{Na}_2\text{S}_2\text{O}_3$  + 10 ml alcohol + neutral soln... yellow ppt.

Conclusion	Confirm tests
ppt. is pot bismuth thiosulphate and radical may be Potassium.	* $\text{Na}_3\text{Co}(\text{NO}_2)_6$ ----yellow ppt. on warning *Sat. tartaric acid soln. ---white ppt. *Perchloric acid-- -- white ppt. *flame test-- Pale violet.

### 9-Test for Na<sup>+</sup>

flame test ----- Golden yellow color

Conclusion	Confirm tests
Radical may be Na.	*NaCo(NO <sub>2</sub> ) <sub>6</sub> ---no ppt. *Tartaric acid----- no ppt. *Pot. Hydrogen antimonite -----white ppt.