

**B.Sc. Programme
(Chemistry Major)**

Laboratory Manual

Semimicro Qualitative Analysis



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QUALITATIVE ANALYSIS
ANALYSIS OF ACID RADICALS

PRELIMINARY REACTIONS

S. No.	Experiment	Observation	Inference
1	Colour and appearance	1) Blue coloured solid 2) Green coloured 3) Brown coloured 4) Puff coloured 5) Pink, coloured 6) Yellow coloured 7) White solid	May be due to the presence of copper May be due to the presence of copper, ferrous ion and nickel May be due to the presence of ferric ion May be due to the presence of manganese May be due to the presence of cobalt May be due to the presence of chromate Absence of coloured salt like Cu, Fe, Mn, Ni, and Co
2.	Solubility a) in water b) in dilute HCl	1) Soluble 2) Insoluble 1) Soluble 2) Insoluble	Presence of water soluble salts Absence of water soluble salts. Absence of I group metals May be due to the presence of I group metal
3	Action of heat : Substance is heated in a dry test tube	1) A colourless gas turning lime water milky is evolved 2) Brown vapours are evolved 3) Violet vapours are evolved 4) A colourless pungent smelling gas fuming with a glass rod dipped in dilute HCl and turning wet red litmus blue is evolved	Presence of carbonate Presence of bromide or nitrate Presence of iodide Presence of ammonium

		5) The residue turns yellow when hot and white when cold	Presence of zinc
		6) No characteristic reaction	Absence of carbonate, iodine, bromide, nitrate, zinc and ammonium
4	<p>Flame Test :</p> <p>a) Substance + Conc. HCl, made into a paste and introduced into the non-luminous part of the flame</p> <p>b) Substance + Conc. H₂SO₄ made into a paste and introduced into the non-luminous part of the flame.</p> <p>c) Boron trifluoride Test : Substance + CaF₂ + conc. H₂SO₄ made into a paste and introduced into the non - luminous part of the flame</p>	<p>1) Bluish green colour is imparted to the flame</p> <p>2) Apple green colour is imparted to the flame</p> <p>3) Crimson red colour is imparted to the flame</p> <p>4) Brick red colour is imparted to the flame</p> <p>5) No characteristic colour is imparted to the flame</p> <p>1) Green colour is imparted to the flame</p> <p>2) No green colour is imparted to the flame</p> <p>1) Green colour is imparted to the flame</p> <p>2) No green colour is imparted</p>	<p>Presence of copper</p> <p>Presence of barium</p> <p>Presence of strontium</p> <p>Presence of calcium</p> <p>Absence of Cu, Ba, Sr and Ca</p> <p>Presence of copper or borate</p> <p>Absence of copper and borate</p> <p>Presence of borate</p> <p>Absence of borate</p>
5	Substance + NaOH warmed	<p>1) A pungent smelling gas fuming with a glass rod dipped in dilute HCl and turning wet red litmus blue is evolved</p> <p>2) No ammonia gas is evolved</p>	<p>Presence of ammonium</p> <p>Absence of ammonium</p>

DRY REACTIONS FOR ACID RADICALS

6	a) The substance is heated with dilute HCl	1) Brisk effervescence takes place in the cold and a colourless gas turning lime water milky is evolved	Presence of carbonate is confirmed
	b) Substance is heated with dil. HCl.	2) No Characteristic reaction	Absence of carbonate
7	The substance is heated with Zn dust and conc. HCl.	1) A colourless gas with the smell of rotten eggs turning lead acetate paper black is evolved	Presence of sulphide
		2) No characteristic reaction	Absence of sulphide
8	The substance is heated with dilute sulphuric acid, cooled well and added a pinch of MnO ₂	1) A colourless gas with a rotten egg smell turning lead acetate paper black is evolved	Presence of sulphide
		2) No H ₂ S gas is evolved	Absence of sulphide
9	The substance is heated with conc. H ₂ SO ₄	1) Brisk effervescence takes place.	Presence of oxalate
		2) No brisk effervescence takes place	Absence of oxalate
		1) Oily drops are seen. A colourless gas forming a white deposit on a wet glass rod is evolved	Presence of fluoride
		2) A colourless gas fuming with a glass rod dipped in ammonium hydroxide is evolved	Presence of chloride
		3) Reddish brown vapours are evolved	Presence of nitrate or bromide
		4) Violet vapours are evolved	Presence of iodide
		5) No characteristic reaction	Absence of nitrate, fluoride, chloride, bromide and iodide

10	The substance is heated. with conc. H_2SO_4 and MnO_2	<p>1) Greenish yellow gas turning starch iodide paper blue is evolved</p> <p>2) Reddish brown vapours turning starch iodide paper blue is evolved</p> <p>3) Violet vapours turning starch paper blue is evolved</p> <p>4) No characteristic gas is evolved</p>	<p>Presence of chloride</p> <p>Presence of bromide</p> <p>Presence of iodide</p> <p>Absence of chloride, bromide and iodide</p>
11	The substance is heated with Conc. H_2SO_4 + Cu turning	<p>1) Reddish brown vapours are evolved</p> <p>2) No reddish brown vapours</p>	<p>Presence of nitrate</p> <p>Absence of nitrate</p>
12	Brown ring test : Substance + conc. H_2SO_4 + heated, cooled and added a drop of freshly prepared ferrous sulphate solution along the sides of the test tube	<p>1) A brown ring is formed</p> <p>2) No brown ring</p>	<p>Presence of nitrate is confirmed</p> <p>Absence of nitrate</p>
13	Ethyl borate test : Substance + conc. H_2SO_4 ethyl alcohol, heated and set fire to the escaping vapours	<p>1) A. green edged flame is seen</p> <p>2) No green edged flame</p>	<p>Presence of borate is confirmed</p> <p>Absence of borate</p>
14	Ammonium molybdate test: The substance is dissolved in dilute HCl and centrifuged. To the centrifugate added a few drops of conc. HNO_3 , cooled well and added excess of ammonium molybdate solution	<p>1) A canary yellow precipitate is got in the cold itself</p>	<p>Presence of phosphate</p>

15	Chromyl chloride test Substance + solid $K_2Cr_2O_7$ + Conc. H_2SO_4 heated	2) An yellow precipitate is got only on heating	Presence of arsenite or arsenate
		3) No canary yellow precipitate is obtained either in cold or on heating	Absence of phosphate, arsenite and arsenate
		1) Reddish brown vapours condensing to a red liquid are obtained	Presence of chloride
		2) No reddish brown vapours	Absence of chloride

WET REACTIONS FOR ACID RADICALS

Preparation of Sodium carbonate Extract :

About 50mg of the substance is mixed with about thrice its amount of solid sodium carbonate and added 5 ml. of distilled water, boiled and centrifuged. The centrifugate is used for the following reactions.

S.No.	Experiment	Observation	Inference
1.	A portion of the extract is acidified with dilute nitric acid, boiled cooled and added silver nitrate solution	1) A curdy white precipitate completely soluble in ammonium hydroxide is got 2) A pale yellow precipitate sparingly soluble in ammonium hydroxide is got 3) A deep yellow precipitate insoluble in ammonium hydroxide is got 4) No characteristic precipitate is formed	Presence of chloride is confirmed Presence of bromide is confirmed Presence of iodide is confirmed Absence of chloride, bromide and iodide
	The above centrifugate is treated with excess of silver nitrate and added, ammonium hydroxide in drops along the sides of the test tube	An yellow ring is got A chocolate coloured ring is got No characteristic ring is got	Presence of phosphate or arsenite Presence of arsenate Absence of phosphate, arsenite and arsenate

2	A portion of the extract is acidified with dilute HCl and added barium chloride solution	A white precipitate insoluble in conc. HCl is formed No white precipitate	Presence of sulphate is confirmed Absence of sulphate
3	A portion of the extract is acidified with dilute acetic acid and added calcium chloride solution The above precipitate is dissolved in hot dilute H ₂ SO ₄ added a few drops of dilute KMnO ₄ solution	A white precipitate is got No white precipitate Permanganate is decoloursied Permanganate is not decolorised	Presence of fluoride or oxalate Absence of fluoride and oxalate Presence of oxalate is confirmed Absence of oxalate and hence presence of fluoride
4	The extract is acidified with dilute HCl and passed H ₂ S gas.	An yellow white precipitate is formed immediately An yellow precipitate is formed after passing H ₂ S for a long time No Yellow precipitate	Presence or arsenite is confirmed Presence of arsenate is confirmed Absence of arsenite and arsenate

Report :

The given mixture contains the acid radicals _____ and _____.

ANALYSIS OF BASIC RADICALS

PREPARATION OF MIXTURE SOLUTION

Major portion of the substance is dissolved in distilled water or dilute HCl or conc. HCl or dilute HNO₃

Elimination of borate / oxalate/ fluoride:

Major portion of the substance is mixed with 5 drops of conc HNO₃ and evaporated to dryness. This process is repeated twice. The dry mass is boiled with dilute HNO₃ and centrifuged.

Elimination of arsenite:

To the I group centrifugate passed H₂S and centrifuged. The yellow precipitate of arsenous sulphide is analysed for II group. Through the centrifugate passed H₂S gas repeatedly, till no more yellow precipitate is formed. The centrifugate is analysed for III, IV, V and VI group.

Elimination of Phosphate: The II group centrifugate is treated with NH₄Cl and Zirconyl chloride. The white precipitate of Zirconium phosphate is discarded.

To the centrifugate added zirconyl chloride repeatedly till no more precipitate is formed. Then centrifuged and the centrifugate is treated with NH₄Cl and NH₄OH and centrifuged. The residue is due to excess of zirconium and III group. The centrifugate is analysed for IV, V and VI groups.

SEPARATION OF BASIC RADICALS INTO GROUPS

To the original mixture solution / eliminated solution added dilute HCl and Centrifuged				
Residue: Presence of I group metals mercurous mercury, silver and lead	Centrifugate : Diluted with water, added dilute HCl passed H ₂ S gas and centrifuged (if arsenite is present, eliminate here)			
	Residue: Presence of II group metals	Centrifugate: A drop of conc. HNO ₃ is added. Boiled to expel H ₂ S gas. Then added the solution of NH ₄ Cl and NH ₄ OH (if phosphate is present, eliminate here)		
	Mercuric mercury, lead, bismuth, copper, cadmium, arsenic, tin and antimony	Residue: Presence of III Group metals iron, manganese, chromium and aluminum	Centrifugate: Added solutions of NH ₄ Cl, NH ₄ OH, passed H ₂ S gas and centrifuged.	
		Residue: Presence of IV group metals cobalt, nickel, manganese and Zinc	Centrifugate: Boiled well to expel H ₂ S gas, the solution is concentrated. Added solution of NH ₄ Cl, NH ₄ OH and (NH ₄) ₂ CO ₃ and centrifuged.	
			Residue: Presence of V group metals barium, strontium and calcium	Centrifugate: Tested for VI group magnesium

ANALYSIS OF GROUP I

The first group precipitate is boiled with a saturated solution of ammonium acetate and centrifuged	
No residue. Absence of mercurous mercury and silver	Centrifugate
	1 .To a portion of the centrifugate added potassium chromate. An yellow precipitate is obtained. Presence of lead
	2. To another portion of the centrifugate added potassium iodide. Yellow precipitate is obtained. Presence of lead
	3.The above precipitate is, boiled with water. the precipitate is dissolved completely and cooled under the tap. Golden yellow spangles are obtained. Presence of lead is confirmed.

ANALYSIS OF GROUP II

The II Group residue is boiled with NaOH solution and centrifuged		
Residue Presence of II A group radicals mercuric mercury, lead, bismuth, copper and cadmium	Centrifugate To the centrifugate added dilute HCl	
	Residue Presence of II B group	No residue Absence of II B group

ANALYSIS OF GROUP II A

Added a few drops of dilute nitric acid and dilute sulphuric acid to the II A group residue, boiled and centrifuged		
No Residue: Absence of mercuric mercury and lead.	Centrifugate Added ammonium hydroxide in drops to excess, heated and centrifuged	
	Residue Added dilute HCl, in drops to dissolve the precipitate then added thiourea solution. Yellow colouration is obtained. Presence of Bismuth	Centrifugate 1. Noted the colour of the centrifugate. Deep blue in colour presence of copper 2. To a portion of the above centrifugate added acetic acid and potassium ferrocyanide. A reddish brown precipitate obtained. Presence of copper is confirmed 3. Another portion of the centrifugate is diluted with water and passed H ₂ S. Yellow precipitate is obtained. Presence of cadmium is confirmed

ANALYSIS OF GROUP II B

The II B group is digested with 5-10 drops of conc. HCl and centrifuged	
<p>Residue : To the residue added saturated $(\text{NH}_4)_2\text{CO}_3$ solution and centrifuged. Acidified the centrifugate with dil. HCl. Yellow precipitate is obtained Presence of arsenic.</p>	<p>Centrifugate : Divided into 2 portions</p> <ol style="list-style-type: none"> 1. To one portion added Zn dust and warmed. Added a few drops of HgCl_2. A silky white precipitate is formed. Presence of tin. 2. To another portion added oxalic acid crystals and passed H_2S gas - A red orange precipitate is got. Presence of Antimony is confirmed

ANALYSIS OF GROUP III

The III group residue is boiled with sodium peroxide and water, stirred and centrifuged	
<p>Residue</p> <p>The residue is dissolved in dil HCl and divided into 2 portions</p> <p>(i) To one portion added few drops of potassium ferrocyanide solution. A deep blue precipitate is obtained. Presence of ion.</p> <p>To find out whether it is ferrous or ferric ion, the original mixture solution is prepared by dissolving the mixture in dil. HCl</p> <p>(a) To one portion of the above solution added potassium ferrocyanide solution. A dark blue solution is obtained. Presence of ferric ion</p> <p>(b) To another portion added potassium ferricyanide solution. A dark blue solution is obtained. Presence of ferrous ion.</p> <p>(ii) To the 2nd portion added dil. HNO_3 and solid sodiumbismuthate - stirred well and centrifuged. A pink centrifugate confirms the presence of Manganese.</p>	<p>Centrifugate</p> <p>Divided into 2 portions.</p> <p>(Noted the colour of the centrifugate yellow colour confirms the presence of chromium)</p> <ol style="list-style-type: none"> 1. To one portion added CH_3COOH and lead acetate solution. Yellow precipitate is obtained. Presence of Chromium 2. To another portion added NaOH in drops to excess. A gelatinous white precipitate soluble in excess of NaOH is obtained. <p>Presence of Aluminum.</p>

ANALYSIS OF GROUP IV

The IV group residue is boiled with dil. HCl and centrifuged		
<p>Residue : To the residue added a few-drops of conc. HCl and KClO₃ of crystals, transferred to a beaker and evaporated to dryness. The drymass is dissolved in, 1ml of distilled water and divided into 2 portions.</p> <p>1) To one portion added solid NH₄CNS and 10 drops of amyl alcohol. A blue alcoholic layer confirms the presence of Cobalt</p> <p>2) To another portion added dimethyl geyoxime and aqueous ammonia A rosy red precipitate is obtained. Presence of Nickel is confirmed.</p>	<p>Centrifugate : Boiled off to expel H₂S gas added NaOH solution to a slight excess & centrifuged</p>	
	<p>Residue The residue is dissolved in dil. HNO₃ & added solid sodium bismuthate. Stirred and centrifuged. A pink colour centrifugate is obtained. Presence of manganese is confirmed.</p>	<p>Centrifugate 1. Through one portion passed H₂S gas A dirty white precipitate is obtained. Presence of Zinc.</p> <p>2 Acidified another portion with CH₃COOH and added potassium ferrocyanide. A bluish white precipitate is formed. Presence of zinc is confirmed.</p>

ANALYSIS OF GROUP V

The V group residue is dissolved in minimum amount of dilute acetic acid, added potassium chromate solution and centrifuged	
Residue Yellow precipitate shows the presence of barium. The above precipitate with conc. HCl imparts apple green colour to the flame. Presence of barium is confirmed.	Centrifugate Neutralised the centrifugate with aq. NH_3 and added ammonium carbonate solution, centrifuged and discarded the centrifugate dissolved the residue in dilute acetic acid and divided into two portions. 1. To one portion added dilute H_2SO_4 . A white precipitate is formed Presence of strontium. The above precipitate with conc. HCl imparts crimson red colour to the flame. Presence of strontium is confirmed. 2. To another portion of the solution added ammonium oxalate and aq. NH_3 solution. White precipitate shows the presence of calcium. The above precipitate is mixed with con. HCl and introduced to a blue flame. Brickred colour is imparted to the flame Presence of calcium is confirmed.

ANALYSIS OF GROUP VI

Test for Magnesium: The VI group centrifugate is evaporated to dryness, dissolved in water and divided into two portions.		
1) To one portion added NH_4Cl , NH_4OH and disodium hydrogen phosphate solutions and scratched the sides of the test tube with glass rod.	A white crystalline precipitate is obtained	Presence of Magnesium
2) To another portion added NaOH solution in drops to excess	A white precipitate insoluble in excess of NaOH is obtained	Presence of Magnesium is confirmed
Test for Ammonium		
1) A portion of the substance is heated with NaOH solution.	A colour gas with pungent smell fuming with a glass rod wetted with conc. HCl is evolved.	Presence of ammonium
2) The mixture is shaken well with distilled water and centrifuged. To the centrifugate added NaOH and Nessler's reagent.	A reddish brown precipitate is obtained.	Presence of ammonium is confirmed

Report :

The given mixture contains the basic radicals _____ and _____.

Result :

Hence the given mixture contains

1. Acid radicals _____ and _____.

2. Basic radicals _____ and _____.

