DIRECTORATE OF DISTANCE & CONTINUING EDUCATIONS

MANONMANIAM SUNDARANAR UNIVERSITY

TIRUNELVELI – 627012

OPEN AND DISTANCE LEARING(ODL) PROGRAMMES

(FOR THOSE WHO JOINED THE PROGRMMES FROM THE ACADEMIC YEAR 2023 – 2024)

B.Sc. CHEMISTRY
COURSE MATERIALS
CORE – VI – QUALITATIVE INORGANIC ANALYSIS

By

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SEMI - MICRO QUALITATIVE ANALYSIS

1. Analysis of simple acid radicals: Carbonate, sulphide, sulphate, thiosulphite, chloride, bromide, iodide, nitrate Analysis of interfering acid radicals: Fluoride, oxalate, borate, phosphate, arsenate, arsenite.
2. Elimination of interfering acid radicals and Identifying the group of basic radicals
3. Analysis of basic radicals (group wise): Lead, copper, bismuth, cadmium, tin, antimony, iron, aluminium, arsenic, zinc, manganese, nickel, cobalt, calcium, strontium, barium, magnesium, ammonium
4. Analysis of a mixture - I to VIII containing two cations and two anions (of which one is interfering type)

Recommended Text Reference Books:

V. Venkateswaran, R. Veeraswamy and A. R. Kulandivelu, Basic Principles of Practical Chemistry, Sultan Chand & Sons, New Delhi, second edition, 1997.

Website and e-learning source

https://www.vlab.co.in/broad-area-chemical-sciences

QUALITATIVE ANALYSIS

ANALYSIS OF ACID RADICALS

PRELIMINARY REACTIONS

S. No.	Experiment	Observation	Inference
1	Colour and appearance	1) Blue coloured solid	May be due to the presence of copper
		2) Green coloured	May be due to the presence of copper, ferrous ion and nickel
		3) Brown coloured	May be due to the presence of ferric ion
		4) Puff coloured	May be due to the presence of manganese
		5) Pink, coloured	May be due to the presence of cobalt
		6) Yellow coloured	May be due to the presence of chromate
		7) White solid	Absence of coloured salt like Cu, Fe, Mn, Ni, and Co
2.	Solubility		
	a) in water	1) Soluble	Presence of water soluble salts
		2) Insoluble	Absence of water soluble salts.
	b) in dilute HCl	1) Soluble	Absence of I group metals
		2) Insoluble	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 HC1 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	Flame Test :		
	a) Substance + Conc. HCl, made into a paste and introduced into the non-luminous part of the flame	1) Bluish green colour is imparted to the flame	Presence of copper
		2) Apple green colour is imparted to the flame	Presence of barium
		3) Crimson red colour is imparted to the flame	Presence of strontium
		4) Brick red colour is imparted to the flame	Presence of calcium
		5) No characteristic colour is imparted to the flame	Absence of Cu, Ba, Sr and Ca
	b) Substance + Conc. H_2SO_4 made into a paste and introduced into the non-luminous part of the flame.	1) Green colour is imparted to the flame	Presence of copper or borate
		2) No green colour is imparted to the flame	Absence of copper and borate
	c) Boron trifluoride Test : Substance + CaF_2 + conc. H_2SO_4 made into a paste and introduced into	1) Green colour is imparted to the flame	Presence of borate
		2) No green colour is imparted	Absence of borate

	the non - luminous part of the flame		
5	Substance + NaOH warmed	1) A pungent smelling gas fuming with a glass rod dipped in dilute HC1 and turning wet red litmus blue is evolved	Presence of ammonium
		2) No ammonia gas is evolved	Absence of ammonium

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
		2) No Characteristic reaction	Absence of carbonate
	b) Substance is heated with dil. 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
7	The substance is heated with Zn dust and conc. HCl.	1) A colourless gas with a rotten egg smell turning lead acetate paper black is evolved	Presence of sulphide
		2) No H_2S gas is evolved	Absence of sulphide
8	The substance is heated with dilute sulphuric acid, cooled well and added a pinch of MnO_2	1) Brisk effervescence takes place.	Presence of oxalate
		2) No brisk effervescence takes place	Absence of oxalate
9	The substance is heated with conc. H_2SO_4	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	1) Greenish yellow gas turning starch iodide paper blue is evolved	Presence of chloride
		2) Reddish brown vapours turning starch iodide paper blue is evolved	Presence of bromide
		3) Violet vapours turning starch paper blue is evolved	Presence of iodide
		4) No characteristic gas is evolved	Absence of chloride, bromide and iodide
11	The substance is heated with Conc. H_2SO_4 + Cu turning	1) Reddish brown vapours are evolved	Presence of nitrate
		2) No reddish brown vapours	Absence of nitrate
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	1) A brown ring is formed	Presence of nitrate is confirmed
		2) No brown ring	Absence of nitrate
13	Ethyl borate test : Substance + conc. H_2SO_4 ethyl alchohol, heated and set fire to the escaping vapours	1) A. green edged flame is seen	Presence of borate is confirmed
		2) No green edged flame	Absence of borate
14	Ammonium molybdate test:	1) A canary yellow precipitate is got in the cold	Presence of phosphate

	The substance is dissolved in dilute HC1 and centrifuged. To the centrifugate added a few drops of conc. HNO_3, cooled well and added excess of ammonium molybdate solution	itself	

		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
15	Chromyl chloride test Substance + solid $K_2Cr_2O_7$ + Conc. H_2SO_4 heated	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	Presence of chloride is confirmed
		2) A pale yellow precipitate sparingly soluble in ammonium hydroxide is got	Presence of bromide is confirmed
		3) A deep yellow precipitate insoluble in ammonium	Presence of iodide is confirmed

		hydroxide is got	
		4) No characteristic precipitate is formed	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	Presence of phosphate or arsenite
		A chocolate coloured ring is got	Presence of arsenate
		No characteristic ring is got	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	Presence of sulphate is confirmed
		No white precipitate	Absence of sulphate
3	A portion of the extract is acidified with dilute acetic acid and added calcium chloride solution	A white precipitate is got	Presence of fluoride or oxalate
		No white precipitate	Absence of fluoride and oxalate
	The above precipitate is dissolved in hot dilute H_2SO_4 added a few drops of dilute $KMnO_4$ solution	Permanganate is decoloursied	Presence of oxalate is confirmed
		Permanganate is not decolorised	Absence of oxalate and hence presence of fluoride
4	The extract is acidified with dilute HCl and passed H_2S gas.	An yellow white precipitate is formed immediately	Presence or arsenite is confirmed
		An yellow precipitate is formed after passing H_2S for a long time	Presence of arsenate is confirmed
		No Yellow precipitate	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 HC1 or conc. HC1 or dilute HNO_3

Elimination of borate / oxalate/ fluoride:

Major portion of the substance is mixed with 5 drops of conc HNO_3 and evaporated to dryness. This process is repeated twice. The dry mass is boiled with dilute HNO_3 and centrifuged.

Elimination of arsenite:

To the I group centrifugate passed H_2S and centrifuged. The yellow precipitate of arsenous sulphide is analysed for II group. Through the centrifugate passed H_2S 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 NH_4Cl 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 NH_4Cl and NH_4OH 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

<table>
<tr><td colspan="6">To the original mixture solution / eliminated solution added dilute Hcl and Centrifuged</td></tr>
<tr><td rowspan="6">Residue: Presence of I group metals mercurous mercury, silver and lead</td><td colspan="5">Centrifugate : Diluted with water, added dilute HCl passed H_2S gas and centrifuged (if arsenite is present, eliminate here)</td></tr>
<tr><td rowspan="5">Residue: Presence of II group metals Mercuric mercury, lead, bismuth, copper, cadmium, arsenic, tin and antimony</td><td colspan="4">Centrifugate: A drop of conc. HNO_3 is added. Boiled toexpel H_2S gas. Then added the solution of NH_4Cl and NH_4OH (if phosphate is present, eliminate here)</td></tr>
<tr><td rowspan="4">Residue: Presence of III Group metals iron, manganese, chromium and aluminum</td><td colspan="3">Centrifugate: Added solutions of NH_4C1, NH_4OH, passed H_2S gas and centrifuged.</td></tr>
<tr><td rowspan="3">Residue: Presence of IV group metals cobalt, nickel, manganese and Zinc</td><td colspan="2">Centrifugate: Boiled well to expel H_2S gas, the solution is concentrated. Added solution of NH_4C1, NH_4OH and $(NH_4)_2C0_3$ and centrifuged.</td></tr>
<tr><td>Residue: Presence of V group metals barium, strontium and calcium</td><td>Centrifugate: Tested for VI group magnesium</td></tr>
</table>

ANALYSIS OF GROUP I

<table>
<tr><th colspan="2">The first group precipitate is boiled with a saturated solution of ammonium acetate and centrifuged</th></tr>
<tr><td rowspan="3">No residue. Absence of mercurous mercury and silver</td><td>Centrifugate
1 .To a portion of the centrifugate added potassium chromate. An yellow precipitate is obtained. Presence of lead</td></tr>
<tr><td>2. To another portion of the centrifugate added potassium iodide. Yellow precipitate is obtained. Presence of lead</td></tr>
<tr><td>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.</td></tr>
</table>

ANALYSIS OF GROUP II

<table>
<tr><th colspan="3">The II Group residue is boiled with NaOH solution and centrifuged</th></tr>
<tr><td rowspan="2">Residue
Presence of II A group radicals mercuric mercury, lead, bismuth, copper and cadmium</td><td colspan="2">Centrifugate
To the centrifugate added dilute HCl</td></tr>
<tr><td>Residue
Presence of II B group</td><td>No residue
Absence of II B group</td></tr>
</table>

ANALYSIS OF GROUP II A

<table>
<tr><td colspan="3">Added a few drops of dilute nitric acid and dilute sulphuric acid to the II A group residue, boiled and centrifuged</td></tr>
<tr><td rowspan="2">No Residue:
Absence of mercuric mercury and lead.</td><td colspan="2">Centrifugate
Added ammonium hydroxide in drops to excess, heated and centrifuged</td></tr>
<tr><td>Residue
Added dilute HC1, in drops to dissolve the precipitate then added thiourea solution. Yellow colouration is obtained. Presence of Bismuth</td><td>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</td></tr>
</table>

		3. Another portion of the centrifugate is diluted with water and passed H_2S. 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	
Residue : To the residue added saturated $(NH_4)_2CO_3$ solution and centrifuged. Acidified the centrifugate with dil. HCl. Yellow precipitate is obtained Presence of arsenic.	Centrifugate : Divided into 2 portions 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	
Residue	**Centrifugate**
The residue is dissolved in dil HCl and divided into 2 portions (i) To one portion added few drops of potassium ferrocyanide solution. A deep blue precipitate is obtained. Presence of ion. To find out whether it is ferrous or ferric ion, the original mixture solution is prepared by dissolving the mixture in dil. HCl (a) To one portion of the above solution added potassium ferrocyanide solution. A dark blue solution is obtained. Presence of ferric ion (b) To another portion added potassium ferricyanide solution. A dark blue solution	Divided into 2 portions. (Noted the colour of the centrifugate yellow colour confirms the presence of chromium) 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. Presence of Aluminum.

is obtained. Presence of ferrous ion. (ii) To the 2nd portion added dil. HNO_3 and solid sodiumbismuthate - stirred well and centrifuged. A pink centrifugate confirms the presence of Manganese.	

ANALYSIS OF GROUP IV

<table>
<tr><th colspan="3">The IV group residue is boiled with dil. HCl and centrifuged</th></tr>
<tr><td>Residue :
To the residue added a few-drops of conc. HCl and $KClO_3$ of crystals, transferred to a beaker and evaporated to dryness. The drymass is dissolved in, 1ml of distilled water and divided into 2 portions.

1) To one portion added solid NH_4CNS and 10 drops of amyl alcohol. A blue alcoholic layer confirms the presence of Cobalt

2) To another portion added dimethyl geyoxime and aqueous ammonia A rosy red precipitate is obtained. Presence of Nickel is confirmed.</td><td colspan="2">Centrifugate :
Boiled off to expel H_2S gas added NaOH solution to a slight excess & centrifuged</td></tr>
<tr><td></td><td>Residue
The residue is dissolved in dil. HNO_3 & added solid sodium bismuthate. Stirred and centrifuged. A pink colour centrifugate is obtained. Presence of manganese is confirmed.</td><td>Centrifugate
1. Through one portion passed H_2S gas A dirty white precipitate is obtained. Presence of Zinc.

2 Acidified another portion with CH_3COOH and added potassium ferrocyanide. A bluish white precipitate is formed. Presence of zinc is confirmed.</td></tr>
</table>

ANALYSIS OF GROUP V

The V group residue is dissolved in minimum amount of dilute acetic acid, added potassium chromate solution and centrifuged	
Residue	**Centrifugate**
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.	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. HC1 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__14_____.