#### KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University)

(Established Under Section 3 of UGC Act 1956)

**Coimbatore – 641 021.** 

#### **SYLLABUS**

#### **DEPARTMENT OF CHEMISTRY**

#### STAFF NAME: Dr.M.R.EZHILARASI

SUBJECT NAME: INORGANIC CHEMISTRY PRACTICAL -I SUB.CODE:18CHP211 SEMESTER: II CLASS: I M.Sc (CHEMISTRY)

Semester-II

# 18CHP211 INORGANIC CHEMISTRY PRACTICAL-I 4H 2C (QUALITATIVE ANALYSIS AND PREPARATIONS)

Instruction Hours/week:L: 0 T:0 P:4 Marks: Internal:40 External: 60 Total:100

**Course Objectives** 

On successful completion of the course the students should have

- 1. Learnt about the qualitative analysis by semi micro-qualitative method.
- 2. Learnt the preparation of inorganic complexes.

#### **Course Outcome**

- 1. Learned about the qualitative analysis by semi micro-qualitative method
- 2. Learned the preparation of inorganic complexes.

#### Contents

Thallium, Tungsten, Selenium, Tellurium, Molybdenum, Cerium, Thorium, Titanium, Zirconium, Vanadium, Beryllium, Uranium and Lithium.



Note: Each student should analyze a minimum of six inorganic mixtures.

#### About ten preparations involving different techniques selected from the following:

Lead tetra acetate, dipyridinium hexaplumbate, hydroxylamine hydrochloride, ortho and parahydroxy phenyl mercuric chloride, potassium cupric chloride, chrome alum, copperI chloride, tris(thio urea) copper(I) Chloride, potassium trioxalato- aluminato(III), potassium trioxalatochromate(III), potassium trioxalato- ferrate(III), hexammine cobalt(III)chloride, chloropentammine chromium(III), chloro aquo pentammine chromium(III) nitrate, tetrammine copper(I) sulphate, ammonium hexa chloro stanate(IV).

Note: Each student should do a minimum of ten preparations.

#### **SUGGESTED READINGS:**

#### **Text Books:**

- 1. Ramanujam, V. V. (2004). *Inorganic Semi-micro Qualitative Analysis* (III Edition). Chennai: The National Publishing Company.
- 2. Venkateswaran, R., Veeraswamy, R. & Kulandaivelu, A.R. (2015). *Basic Principles of Practical Chemistry*. New Delhi: S. Chand & Sons Ltd.
- 3. Siddhiqui, Z. N. (2002). *Practical Industrial Chemistry* (I Edition). New Delhi: Anmol Publications Pvt. Ltd.

#### **Reference Books:**

- 1. Mendham, J. R., Denney, C., Barnes, J. D., & Thomas, M. (2002). *Vogel's Textbook of Quantitative Chemical Analysis* (VI Edition). Singapore: Pearson Education Ltd.
- 2. Lepse, P. A., & Peter, L. B. (1986). *Lab Manual for Lingren's Essentials of Chemistry*. New Delhi: Prentice Hall.



#### **KARPAGAM ACADEMY OF HIGHER EDUCATION**

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#### **LECTURE PLAN**

#### **DEPARTMENT OF CHEMISTRY**

Staff Name	: Dr.M.R.Ezhilarasi
Subject Name	: Inorganic Chemistry Practical -I
Sub Code	·18CHD211

Sub.Code :18CHP211

Semester

: II Class : I M.Sc (Chemistry)

#### **Total Lecture Hours: 40 Hours**

S.No	Lecture Duration Period	Topics to be Covered	Support Material/Page Nos
1	4	Preparation of chrome alum, tris(thio urea) copper(I) Chloride,	T1:73
2	4	Preparation of tetrammine copper(II)	T2:501
		chromate(III)	T1:72-73, 74
3	4	Preparation of potassium trioxalato-	T2:505
		ferrate(III), potassium trioxalato- aluminato(III)	T1:74-75
4	4	Preparation of copperI chloride, lead tetra acetate	T1:78
5	4	Preparation of ortho and para- hydroxy phenyl mercuric chloride, hydroxylamine hydrochloride	T1:76-77

6	4	Analysis of inorganic mixture-I	T2:41-66
7	4	Analysis of inorganic mixture-II	
8	4	Analysis of inorganic mixture-III	T2:41-66
9	4	Analysis of inorganic mixture-IV	T2:41-66
10	4	Model Practical examination	
	Total No of Hou	rs Planned For Practical= 40 hours	

#### **REFERENCE BOOK**

T1: V. Venkateswaran, R. Veerasamy and A.R. Kulandaivelu, 2015, Basic Principles of Practical Chemistry, Sultan Chand & Sons Publishers, NewDelhi.

T2: A.O. Thomas, 2003, Practical Chemistry for B.Sc Main Students, Scientific Book Centre, Cannore-1, Kerala

# **DEPARTMENT OF CHEMISTRY**

# PRACTICAL MANUAL

## **INORGANIC CHEMISTRY-I PRACTICAL**



Karpagam Academy of Higher Education (Deemed to be University) Established Under Section 3 of UGC Act, 1956) Pollachi Main Road,Eachanari Post, Coimbatore – 641 021,Tamil Nadu,India.

# LIST OF PRACTICALS

S. NO.	TITLE OF EXPERIMENTS	PAGE NO.
1.	PREPARATION FOR CHROME ALUM (K <sub>2</sub> SO <sub>4</sub> .Cr <sub>2</sub> SO <sub>4</sub> .24H <sub>2</sub> O)	3
2.	PREPARATION OF TRIS [thio-urea] COPPER I CHLORIDE	4
3.	PREPARATION OF TERA AMINE COPPER(III) SULPHATE	5
4.	PREAPRATION OF POTASSIUM TRIOXALATO CHROMATE (III)	6
5.	PREPARATION OF POTASSIUM TRIOXALATE FERRATE(III)	7
6.	PREPARATION OF POTASSIUM TRIOXALATE ALUMINATE(III)	8
7.	PREPARATION OF COPPER (I) CHLORIDE	9
8.	PREPARATION OF HEXAMMINE COBALT(III) CHLORIDE	10
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#### 1)PREPARATION FOR CHROME ALUM(K2SO4.Cr2SO4.24H2O)

#### **CHEMICALS REQUIRED:**

 $Con.H_2SO_4 = 1.5ml$ 

Potassium Dichromate =15g

#### **PROCEDURE:**

In a 400ml beaker 12.5ml of conc.  $H_2SO_4$  and 11.5ml of water is taken and 15g of  $K_2Cr_2O_4$  powder is added. It is cooled in a ice cold water bath and added 7ml of alcohol with constant stirring. A temperature should be maintained at or near 50°Cif necessary depend of piece of ice in a mixture. The solution is concentrated in water bath below 60°c. The solution is cooled in a ice bath. The content is allowed to stand and crystals are collected. The product obtained is recrystallized from cold water.

#### **RESULT:**

The yield of the product:

#### 2) PREPARATION OF TRIS [thio-urea] COPPER I CHLORIDE

#### **CHEMICALS REQUIRED:**

Cupric chloride dehydrate	-2.7g
Thio urea	-5g
Con .HCL	-5ml

#### **PROCEDURE:**

Dissolved 5g of thiourea in 30ml of hot water. To the solution added 2.7g cupric chloride dehydrate and 5ml con .HCL in 15 ml of deionized water. Heat on a boiling water bath, filtered the hot solution and allowed the clear solution to cool and filtered. The product recrystallized the sample from 5% aqueous thiourea solution or with small amount of dil.HCl.

#### **RESULT:**

#### 3) PREPARATION OF TERA AMINE COPPER(III) SULPHATE

#### **CHEMICALS REQUIRED:**

Coppersulphatepentahydirde	-5g
Concentrated ammonia	-7.5g
Ethanol	-10ml

#### **PROCEDURE:**

Dissolved 5g of copper sulphatepentahydrate in 10ml of deionized water. Added 7.5ml of conc.ammonia and the resulting solution is filtered. The above clear solution is taken in a clean beaker about 10ml of alcohol is added and allowed the solution to cool. The crystals formed are filtered and washed with 1:1 alcohol, ammonia solution and dried. The complex is recrystallized from 1:1 alcohol water mixture.

#### **RESULT:**

#### 4) PREAPRATION OF POTASSIUM TRIOXALATO CHROMATE (III)

#### **CHEMICALS REQUIRED:**

Oxalic acid	-3g
Potassium dichromate	-1g
Potassium oxalate	-1.3g

#### PROCEDURE:

To a solution of 3g of oxalic acid in 20ml of warm water, added in portion, 1g of potassium dichromate. Heated the solution to boiling and dissolved 1.3g of potassium oxalate in the solution. Then cooling is continued until the blue green crystal separated. The sample is recrystallized by using 1:1 water alcohol mixture.

#### **RESULT:**

#### 5)PREPARATION OF POTASSIUM TRIOXALATE FERRATE(III)

#### **CHEMICALS REQUIRED:**

Ferrous oxalate	-5g
Potassium oxalate	-5g
Hydrogen peroxide	-15ml
Oxalic acid	-15ml(105%)
Ethanol	-10ml

#### **PROCODURE:**

Suspended 5g of ferrous oxalate in warm solution of 5g of potassium oxalate in 30ml of water 15ml of hydrogen peroxide is added from the burette. The solution is stirred continuously during the addition and the temperature is kept at 40°C then heated the mixture to boiling and the precipitate is dissolved by adding 10ml of 10% oxalic acid. Then added 3ml of excess during the addition of oxalic acid the liquid should be nearly boiling. Filter the solution and added 10ml of ethanol to the filtrate. Then it is gently heated in a water bath on cooling the complex crystals. The precipitate recrystallized using 1:1 water alcohol mixture.

#### **RESULT:**

#### 6)PREPARATION OF POTASSIUM TRIOXALATE ALUMINATE(III)

#### **CHEMICALS REQUIRED:**

Aluminium powder	- 0.5g
Potassium hydroxide	- 20g of 100ml
Oxalic acid	- 7g

#### **PROCEDURE:**

Weighed about 0.5g of concentrated aluminium powder into a 400ml beaker.Added heated to boiling to dissolved aluminum filtered. The solution and added 10ml of water to the filtrate and the solution heated to boiling weighed about 7g of oxalic acid and added to boiling solution. The solution filtered and cooled. When the solution gets the room temperature added about 10ml of ethanol and the cooling continued. The formed crystals are separated by filtration and dried. The crude sample is recrystallized using water as solvent.

#### **RESULT:**

#### 7)PREPARATION OF COPPER (I) CHLORIDE

#### **CHEMICALS REQUIRED:**

Copper sulphatepentahydrate	-10g
Sodium chloride	-5g
Copper turnings	-10g
HCl	-30ml
Sodium sulphate	-4g
Alcohol, ether	-4g

#### **PROCEDURE:**

10g of copper sulphate pentahydrate,5g of NaCl and 10g copper turnings are mixed ultimately and then placed in a 250ml conical flask about 50ml of con.HCl is added to the flask and the mixture is heated in a water bath. For nearly over hour until the blue or bluish green color disappearance completely. When the solution in the flask acquires a stain color it is poured in to beaker containing about 500ml of water containing about 3 to 4g of sodium sulphate. A dull white precipitate of cuptous chloride settles to the bottom immediately it is filtered by a buckner funnel. It is washed with alcohol and finally with ether and dried in a desicator.

#### **RESULT:**

#### 8) PREPARATION OF HEXAMMINE COBALT(III) CHLORIDE

#### AIM:

To preparation of hexamine cobalt (III) chloride from ammonium chloride and cobaltous chloride.

#### **PRINCIPLE:**

Preparation of hexammonium cobalt (III) chloride involves the mixing of ammonium chloride and cobaltous chloride followed by the addition of con.ammonia and hydrogen peroxide.

$2CoCl_2+2NH_4Cl$	+10NH <sub>3</sub>	>	$2[Co(NH_3)_6]Cl_3$	+	$2H_2O$
	11011113	~			

#### **CHEMICALS REQUIRED:**

Ammonium chloride	-12g
Cobaltous chloride	-18g
20 volume hydrogen peroxide	-35m

#### PROCEDURE:

18g of cobaltous is dissolved in a boiling solution of 12g of ammonium chloride in 25 ml of water. Add 1g of animal charcoal and cool the contents in the running water. Wash out the vessel in which the solution was first made with 40ml of conc. ammonia and add the ammonical liquid to the flask. Cool the whole contents of preparation in icebath to 10°c. 35ml of 20 volume hydrogen peroxide is added slowly in portion while shaking the flask and its contents till the oxidizing having been added, heat the mixture by shaking until the portion in the liquid is removed crystals being to separate quantity on cooling in aice bath.Filter on the crude solid and without washing it, transfer into a beaker containing a boiling mixture of 150ml of water and 5ml

of con.HCl. when all solid except the charcoal has dissolved filter the liquid an ice bath. Golden brown crystals separate out.

#### **RESULT:**

Yield of the product:

#### 9) PREPARATION OF AMMONIUM HEXACHLORIDE STANNATE(IV)

#### AIM:

To prepare ammonium hexachloridestannate(IV) from tin and ammonium chloride.

#### **PRINCIPLE:**

Stannous(II)chloride solution is oxidized to stannic IV solution dry heating it with con.nitric acid upon adding ammonium chloride to this solution the complex, ammonium hexachlorostannate(IV)is obtained.

#### **CHEMICALS REQUIRED:**

Tin-10gAmmonium chloride-9gConc.Nitric acid-00Conc. HCl-00

#### PROCEDURE:

10g of the tin is placed in a flask and warmed with con.Hcl until the metal negins to dissolved displacing hydrogen then add con.nitric acid is sufficient quantity to oxidize the stannous (II) to stannic(IV) state. When the metal has dissolved, test a few drops of the solution with HgCl<sub>2</sub>. If a white precipitate is formed stannous tin is presence and a little more con.nitric acid must be added to oxidize it filter the stannic solution. 9g of ammonium chloride is dissolved in small quantity of hot water mix these two solution and evaporate until on cooling ammonium hexachlorostannate(IV) crystallizes out from the solution filter at a pump wash with a little con.HCl acid and dry it.

#### **RESULT:**

The amount ammoniumhexachlorostannate(IV)=

# 10)PREPARATION OF ORTHO AND PARA HYDROXYL PHENYL MERCURIC CHLORIDE

#### AIM:

To prepare ortho and para hydroxyl phenol mercuric chloride.

#### **PRINCIPLE:**

By heating a mixture of phenol and mercuric acetate in presence of sodium chloride two isomers are obtained. The ortho isomer melts sharply at 152 and concise of a mass of very small needles.

The folloeingreaction are involved in the preparation.

 $C_6H_5OH+Hg(CH_3COO)_2---->HO-C_6H_4-Hg(CH_3COO)+CH_3COOH$ 

HO-C<sub>6</sub>H<sub>4</sub>+Hg(CH<sub>3</sub>COO)+NaCl---->HO-C<sub>6</sub>H<sub>5</sub>HgCl+CH<sub>3</sub>COONa

#### **CHEMICALS REQUIRED:**

Phenol	-3g
Mercuric acetate	-5g
Sodium chloride	-1.25g

#### PROCEDURE:

30g of phenol is melted in a beaker of 150ml capacity by setting the beaker with contents on the boiling water bath add 5g of dry mercuric acetate in portions when all the solid has dissolved add 75ml of boiling water and stirred the mixture boiling the contents of the beaker over a small free flame and it necessary and continuously a small amount of boiling water until or vigorous shaking the oil dissolves leaving only a small residue. The residue usually becomes firmly

attached to the walls of the beaker and the color solution can readily be decanted from it an dissolving 1.25g of the solution sodium chloride in the hot solution its para isomer at on separates and is reduced granular by keeping it on boiling water bath for 10 min. After filtering p-isomer from the hot liquid. The other isomer crystallizes from the cooling filtrates the p-isomer a washed on the filter with hot water and more soluble ortho isomer with small quantity of cold water.

#### **RESULT:**

Amount of the two isomers obtained:

O-compound=

P-compound=

# **INORGANIC QUALITATIVE ANALYSIS**

EXPERIMENT	OBSERVATION	INFERENCE
1. <u>Colour and appearance</u>	1. Blue or green	Presence of copper
	2. Pink	Presence of cobalt,
	3. Green	manganese
	4. Colourless solid	Presence of manganese
		Absence of copper,
		manganese and cobalt
2. <u>Solubility</u>		
a) In water	Soluble	May be ammonium salts
	Insoluble	Absence of ammonium
		salts
b) In dil. HCl	Soluble	
	Insoluble	Absence of lead
		May be due to lead
3. Flame colour test:		
A pinch of given substance is made	1. Bright green	Presence of borate
into paste with 2 or 3 drops of conc.	2. Bluish green	Presence of copper
HCl in a watch glass. The paste is	3. Light Yellowish green	Presence of barium
taken at the end of a charred splinter	4. Brick red	Presence of calcium
and introduced into the edge of non-	5. No characteristic	Absence of borate, copper,
luminous flame. The colour of the	coloured flame	barium and calcium
flame is noted.		
4. The flame test is performed with	A bright green colour is	Presence of borate
the substance and conc. H <sub>2</sub> SO <sub>4</sub> .	imparted	Absence of borate
	No bright green coloured	
	flame	
5. Boron trifluoride test:		
A pinch of the substance is mixed	A bright green colour is Presence of borate	
with calcium fluoride and a drop of	of imparted to the flame.	
$conc.H_2SO_4$ is added and made into a		

paste. It is taken at the end of charred	No bright green colour is	Absence of borate
splinter and introduced into the edge	imparted to the flame.	
of non-luminous flame.		
6. <u>Ethyl Borate test</u> :		
A pinch of substance is mixed with	The vapour burns with a	Borate confirmed.
2cc of ethyl alcohol and 10 drops of	green edged flame.	
conc.H <sub>2</sub> SO <sub>4</sub> in a test tube. It is heated		
and the vapour is ignited.	The vapour does not burn	Borate is absent.
	with green edged flame.	
7. <u>Action of heat:</u>		
A small portion of the substance is	1. Colourless gas turning	Presence of carbonate and
heated in a dry test tube.	lime water milky.	oxalate
	2. Colourless gas with smell	Presence of ammonium
	of NH <sub>3</sub> and fuming with	salts
	conc. HCl is evolved.	
	3. Red brown gas turning	
	ferrous sulphate paper	Presence of nitrate
	brown.	
	4. White sublimate is	
	formed.	Presence of ammonium
	5. Yellow when hot white	salts
	when cold.	Presence of zinc
	6. No characteristic change.	
		Absence of carbonate,
		oxalate, ammonium,
		nitrate and zinc
8. Action of sodium hydroxide:		
A pinch of substance is boiled with 5	Colourless pungent smelling gas	Presence of ammonium
cc of NaOH solution.	is evolved. It turns red litmus blue	
	and gives dense white fumes with	

	glass rod dipped in conc. HCl.	
	No characteristic gas	Absence of ammonium
9. Action of dil. HCl:		
A pinch of the substance is treated	1. Brisk effervescence of	Carbonate is confirmed.
with 3 cc of dil. HCl in a test tube.	colourless gas is evolved.	
	It turns lime water milky.	
	2. Colourless gas with the	
	smell of rotten egg	Sulphide is confirmed.
	turning lead acetate paper	
	black.	
	3. No characteristic change	
		Absence of carbonate and
		sulphide.
10. Action of dil. H <sub>2</sub> SO <sub>4</sub> and		
MnO <sub>2</sub> :	Effervescence takes place and	Presence of oxalate
To a pinch of the substance, a pinch	colourless gas is evolved turning	
of $MnO_2$ and 3 cc of dil. $H_2SO_4$ are	lime water milky.	
added and warmed gently		
	No Effervescence takes place	Absence of oxalate
11. Action of Conc. H <sub>2</sub> SO <sub>4</sub> :		
A pinch of the substance is heated	1. Colourless gas giving	Presence of chloride
with 2 cc of conc. H <sub>2</sub> SO <sub>4</sub>	dense white fumes with	
	ammonia is evolved.	
	2. Colourless gas giving	Presence of fluoride
	white precipitate with a	

	glass rod dipped in water.	
	Oily appearance at the	
	top of the test tube.	
	3. Yellowish brown vapour	
	evolves on continued	Presence of nitrate
	boiling and no action of	
	fluorescence paper	
	nuorescence paper.	
	4. A colourless gas evolves	
	turning lime water milky.	Presence of oxalate
	5. No characteristic gas is	Absence of chloride,
	evolved	fluoride,
		nitrate and oxalate
12 Togt for halidage		
12. <u>Test for handes</u> :		
A pinch of the substance is mixed	1. Colourless gas evolves	Presence of oxalate
with a pinch of $MnO_2$ and 2 cc of	which turns lime water	
conc. H <sub>2</sub> SO <sub>4</sub> and warmed gently	milky.	
	2. Greenish yellow gas	Presence of chloride
	evolves which turns	
	starch iodide paper blue	
	2 Ded brown cas has no	
	3. Red blown gas has no	
	action with fluorescence	Presence of nitrate
	paper.	
	4. No characteristic change	Absence of oxalate,
		chloride and nitrate
13. <u>Chromyl chloride test:</u>		
A pinch of the substance is warmed	Red brown gas is evolved	Presence of chloride
with a few amount of potassium	forming vellow precipitate with	
diabromate and cone U.SO	load agatata solution	
dichromate and conc. H <sub>2</sub> SO <sub>4</sub> .	lead acetate solution.	

	No red brown gas	Absence of chloride.
14. <u>Test for nitrate:</u>		
A pinch of substance is heated with 2	Red brown gas turning ferrous	Presence of nitrate.
c c of conc. $H_2SO_4$ and pieces of	sulphate paper brown.	
copper turning are added and		
warmed.	No red brown gas is seen.	Absence of nitrate.
15. <u>Ammonium molybdate test:</u>		
A pinch of the substance is dissolved	A bright yellow precipitate at	Presence of phosphate.
in few drops of conc. $HNO_3$ and the	once or gradually in the cold.	
clear solution is heated with 5-10 cc		
of ammonium molybdate solution,	No yellow precipitate	Absence of phosphate.
shaken well and heated gently		
16. Action of conc. HCl and tin:		
A little of the substance is heated	Colourless rotten egg smell gas	Presence of sulphide
with a pinch of tin metal and a few	turning lead acetate paper black	
drops of conc. HCl.		
	No rotten egg smell	Absence of sulphide.

#### Preparation of sodium carbonate extract:

A little of the given substance is mixed with three times of sodium carbonate and one test tube of distilled water in a boiling tube and the contents are boiled for ten minutes. The solution is filtered and the filtrate is used for the following reactions.

1. Barium Chloride Test:		
To about 1 cc of the extract, dil.	A white precipitate	Sulphate is confirmed.
HCl is added drop by drop till the	insoluble in conc. HCl is	
effervescence stops. The contents	formed.	
are heated, cooled and barium		
chloride solution is added.	No white precipitate is	Absence of sulphate.
	formed.	

2. <u>Silver Nitrate Test:</u>		
To about 1 cc of the extract, dil.	A curdy white precipitate	Chloride is confirmed.
HNO <sub>3</sub> is added drop by drop till the	soluble in ammonium	
effervescence stops. The contents	hydroxide is formed.	
are heated, cooled and Silver nitrate		
solution is added.	No curdy precipitate is	Absence of chloride.
	formed.	
If no precipitate on adding AgNO <sub>3</sub> ,		
then few drops of ammonium	A yellow precipitate is	Presence of phosphate
hydroxide are added drop by drop.	obtained in the form of a	
	ring.	
	No characteristic ring is	Absence of phosphate
	seen	
3. About 1 cc of the extract is	A yellow precipitate is	Presence of phosphate
boiled with few drops of conc.	formed on cold or on	
$HNO_3$ and cooled. Then it is	gentle warming	
shaken with 10 cc of		
ammonium molybdate solution.	No yellow precipitate	Absence of phosphate
	precipitate	
4. <u>Calcium chloride Test:</u>		
About 2 cc of the extract is	A white precipitate is	Presence of fluoride or
acidified with acetic acid, boiled	obtained	oxalate.
with 5 cc of calcium chloride		
solution.		
If there is a white precipitate	1. KMnO <sub>4</sub> solution is	Oxalate is confirmed.
obtained in the above experiment, it	decolourised.	
is filtered off. The white precipitate		

is dissolved in 3 cc of dil. H <sub>2</sub> SO <sub>4</sub> ,		
heated and to the clear solution, a	2. KMnO <sub>4</sub> solution is not	Fluoride is confirmed
dil. solution of KMnO4 is added	decolourised.	
drop by drop.		
5. Brown Ring Test:		
About 2 cc of the extract is	A brown ring is formed at	Nitrate is confirmed.
acidified with dil. $H_2SO_4$ and the	the junction of the two	
mixed with 3 cc of strong ferrous	liquids.	
sulphate solution. Conc. H <sub>2</sub> SO <sub>4</sub> is		
added along the sides of the tube.	No brown ring is formed.	Absence of nitrate.

#### **Removal of Interfering radicals:**

#### **<u>1. Elimination of borate and fluoride:</u>**

A portion of the given substance is mixed with 5 cc of conc. HCl in a boiling tube. The mixture is evaporated to a paste and cooled. 5 cc of conc. HCl is added and again evaporated to a paste. The process is repeated four times. The paste is finally mixed with 10 cc of water. If any precipitate is formed, it is used for the analysis of I group cations. The solution is used for the analysis from II group.

#### 2. Elimination of oxalate:

A portion of the given substance is roasted strongly in a china dish for 20 minutes. The mass is then cooled and mixed with 10 cc of conc. HCl. The mixture is boiled and filtered. The filtrate is evaporated to a paste. The paste is finally mixed with 10 cc of water. If any precipitate is formed, it is used for the analysis of I group cations. The solution is used for the analysis from II group.

#### 3. Elimination of phosphate:

The phosphate radical is eliminated before proceeding to group III. A few drops of the filtrate from group II, after the removal of  $H_2S$ . To the neutral solution, zirconyl chloride (ZrOCl<sub>2</sub>) is added and the precipitate got is removed. The process is repeated till no precipitate is obtained.

### ANALYSIS OF BASIC RADICALS

#### **Separation of cations into groups**

A small amount of the substance is dissolved in water, dil. HCl or conc. HCl (The residue should examined for I group)

To 1 cc of the original solution, dil.HCl is added and centrifuged.

Residue:	Centrifugate: T	o the filtrate H <sub>2</sub> S	gas is passed and	d centrifuged.	
Presence of I	Characteristic Residue:	<b>Centrifugate:</b> It is boiled to expel $H_2S$ and then added 2 drops of conc. HNO <sub>3</sub> and boiled with ammonium chloride and excess of ammonium hydroxide and centrifuged			
group	group	Characteristic Residue:	Centrifugate: I	$H_2S$ is passed and centri	fuged
		Presence of III group	Characteristic Residue: Presence of IV group	Centrifugate:BoiledBoiledwithdconcentrated to reducThenammoniumammoniumhydroammoniumammoniumcarbonatand centrifuged.Residue:Presence of V group	to expel H <sub>2</sub> S. il. HNO <sub>3</sub> , e the volume. chloride, oxide and e are added Centrifugate: Analysed for VI group

### **DETECTION OF CATIONS: ANALYSIS OF I-GROUP**

The white residue is washed, boiled with water and centrifuged while hot.

Residue: Shaken with ammonium hydroxide		Centrifugate:
and centrifuged		i) To one part, KI solution and water
Residue: Black. 3 drops of	Centrifugate:	are added. A small amount of the
conc. HCl and 1 drop of	Dil. HNO <sub>3</sub> is	yellow precipitate is dissolved by
conc, HNO <sub>3</sub> are added and	added. White	heating and cooled. Golden spangles
centrifuged. To the clear	precipitate. Ag <sup>+</sup>	appear. Lead is confirmed.
Centrifugate, stannous	is confirmed.	ii) To the second part, potassium
chloride is added. White		chromate is added. Yellow precipitate.
silky precipitate turning		Lead is confirmed.
grey. Hg <sup>2+</sup> is confirmed.		

### **ANALYSIS OF II A GROUP**

The residue is washed with 1 cc of water, 1 cc of dil.  $HNO_3$  and 1 cc dil  $H_2SO_4$  are added boiled and centrifuged.

No residue	Centrifugate: Ammonium hydroxide is added in drops to excess, boiled		
Absence of	and centrifuged.		
Absence of	Residue: It is	Centrifugate: Divided into two portions.	
mercury and	dissolved in dil.	1. To one portion, a few drops of acetic acid is added	
lead. HCl and poured		and followed by potassium ferrocyanide. Red brown	
	into excess	precipitate. Copper is confirmed.	
	water. White	2. To another portion, a small amount of water and	

turbidity.	H <sub>2</sub> S gas is passed. Yellow precipitate is formed.
Presence of	Cadmium is confirmed.
bismuth.	

# ANALYSIS OF III GROUP

The residue is washed with water, boiled with water and centrifuged.

Residue: Brown	Centrifugate: Dil.
1. One part is dissolved in dil. HCl and the solution is divided into two	HCl, 1 g of solid
parts.	ammonium chloride
a) To one part potassium ferricyanide is added. Prussian blue precipitate.	are added.
Ferric ion is confirmed.	Gelatinous white
b) To another part potassiumthiocyanate is added. Blood red colour. Ferric	precipitate.
ion is confirmed.	Aluminium is
2. The second part of the precipitate is boiled with conc. $HNO_3\&$ a pinch	confirmed.
of lead oxide or sodium bismuthate is added, diluted and allowed to stand.	
Pink colour of permanganate is obtained. Manganese is confirmed.	

# **ANALYSIS OF IV GROUP**

The residue is washed with water and stirred with dil. HCl and centrifuged.

Residue: Black. It is boiled with	Centrifugate: Boiled to expel H2S and slight
	excess NaOH is added and centrifuged.

conc.HCl& a crystal of potassium chlorate	Residue:	Centrifugate:
and evaporated to dryness. Blue residue –	1. One part is boiled	1. Through one part, H <sub>2</sub> S is
	with PbO <sub>2</sub> and	passed. White precipitate-
cobalt; yellow residue – nickel. It is	conc. HNO <sub>3</sub> and	zinc is confirmed.
dissolved in water.	diluted. Pink colour	2. To another part, acetic
1. To one part, ammonium thiocyanate and	– Manganese is	acid and potassium
amy alashal are added and shaken well	confirmed.	ferricyanide are added.
any aconor are added and shaken wen-	2. Another part is	White precipitate- zinc is
alcohol layer is blue. Cobalt is confirmed.	fused with KOH	confirmed.
2. To another part, ammonium chloride,	and KClO <sub>3</sub> , green	3. The precipitate in (1) is
ammonium hydraoxide and dimethyl	residue turns pink.	dissolved in conc. HNO <sub>3</sub> , 2
	On adding dil.	drops of cobalt nitrate is
glyoxime are added. A rosy red precipitate	H <sub>2</sub> SO <sub>4</sub> confirms	added. A filter paper dipped
- nickel is confirmed.	manganese.	in the solution is burnt to
		ash. Green ash-Zinc is
		confirmed.

# ANALYSIS OF V GROUP

The residue is washed with water and dissolved in minimum amount of acetic acid, potassium chromate is added and centrifuged.

Yellow Residue: Barium is confirmed.	Centrifugate:	
Dissolved in conc. HCl. Flame colour test is	1. To this, ammonium chloride, ammonium	
performed. Green coloured flame-barium is	hydroxide and ammonium oxalate solutions are	
confirmed.	added. White precipitate – Calcium is	
	confirmed.	
	2. Flame colour test is performed with the	
	precipitate. Brick red coloured flame. Calcium	
	is confirmed.	
	is confirmed.	

## ANALYSIS OF VI GROUP

Centrifugate from V group is concentrated to a small volume by evaporation and divided into two portions.

1. To one part, ammonium chloride, ammonium hydroxide and disodium hydrogen phosphate are added. The inner sides of the test tube is scratched with a glass rod. White crystalline precipitate. Magnesium is confirmed.

2. To the second part, sodium hydroxide is added in excess. White precipitate insoluble in excess of NaOH confirms magnesium.

#### Test for ammonium radical:

1. Heat a little mixture with 2-3 cc of NaOH in a test tube. A characteristic smell of ammonia is obtained. On bringing a glass rod dipped in conc. HCl on the mouth of the test tube, enormous white fumes are produced, ammonium is confirmed.

On adding Nessler's reagent to the solution of the mixture – Brown colour or precipitate.
 Ammonium is confirmed.

#### **<u>Report:</u>**

The given inorganic mixture contains Acid Radicals: 1)

2)

Basic Radicals: 1)

2)