KARPAGAM ACADEMY OF HIGHER EDUCATION
(Deemed to be University Established Under Section 3 of UGC Act 1956)
Coimbatore – 641 021.

SYLLABUS
DEPARTMENT OF CHEMISTRY
M.Sc CHEMISTRY

Semester - II

17CHP211 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

Scope

This practical deal with the semi micro-qualitative analysis and spot tests of mixtures of familiar cations and non familiar cations and to motivate the students to understand the basic principles of lab techniques adopted in laboratories.

Objectives

On successful completion of the course the students should have

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

Methodology

Blackboard teaching and Demonstration.

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 para-hydroxy phenyl mercuric chloride, potassium cupric chloride, chrome alum, copperI chloride, tris(thio urea) copper(I) Chloride, potassium trioxalato- aluminato(III), potassium trioxalato-chromate(III), potassium trioxalato- ferrate(III), hexammine cobalt(III)chloride, chloropentammine chromium(III), chloro aquo pentammine chromium(III) nitrate, tetrammine copper(II) 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 Analysi* (III Edition). Chennai: The National Publishing Company.
2. Venkateswaran, V., Veeraswamy, R., & Kulandaivelu, A. R. (2004). *Basic Principles of Practical Chemistry* (II Edition). New Delhi: S. Chand Publications.
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.

INORGANIC CHEMISTRY LABORATORY

MANUAL

QUALITATIVE ANALYSIS OF INORGANIC MIXTURES AND PREPARATIONS

CONTENTS

***Reference*s Books**:

R1. Ramanujam, V.V. 1993. Inorganic Semi-micro Qualitative Analysis. III Edition, The National Publishing Company, Chennai (2004).

R2. V.Venkateswaran, R.Veeraswamy and A.R.Kulandaivelu, Basic Principles of Practical Chemistry, 2nd Edition, S.Chand Publications, New Delhi (2004).

QUALITATIVE ANALYSIS OF INORGANIC MIXTURES

PREPARATION OF ORIGINAL SOLUTION

A small quantity of the given mixture is treated with 10 ml of dil.HNO_3 containing few drops of con.HNO_3. It is boiled and cooled. Added 2 ml of water to dilute the solution.

SEPARATION OF CATIONS INTO GROUPS

To the original solution added 10 drops of Con.HCl boiled, cooled and centrifuged.

<table>
<tr><td>Residue</td><td colspan="4">Centrifugate Added a small quantity of hydrazine hydrochloride, boiled and centrifuged.</td></tr>
<tr><td></td><td>Residue</td><td colspan="3">Centrifugate Added a drop of 6% H_2O_2 and boiled. Diluted with 3ml of water. Added 1ml of dilute HCl and boiled. Passed H_2S gas or added yellow ammonium sulphide solution.</td></tr>
<tr><td></td><td></td><td>Residue</td><td colspan="2">Centrifugate Boiled of H_2S gas. Added 2 drops of con.HNO_3 and boiled. A drop of this solution is tested for ferric iron. (Solution +NH_4CNS=Blood Red Colour). To the remaining solution added 2 drops of $FeCl_3$ and 5 drops of NH_4Cl. Boiled to the hot solution added NH_4OH in excess and centrifuged.</td></tr>
<tr><td></td><td></td><td></td><td>Residue</td><td>Centrifugate Added NH_4OH and passed H_2S gas or yellow ammonium sulphide solution. Boiled and centrifuged.</td></tr>
<tr><td></td><td></td><td></td><td>Residue</td><td>Centrifuged Neutralized with dilute HNO_3 solution concentrated. Added excess of NH_4OH and $(NH_4)_2CO_3$. Boiled and centrifuged.</td></tr>
</table>

I GROUP	**IAGROUP**	**II GROUP**	**IIIGROUP**	**IV GROUP**	**Residue** **V GROUP**	**Centrifuged** Tested separately for Magnesium, Lithium and Ammonium ions. **VI GROUP**

ANALYSIS OF GROUP-I

To the I group residue added 3ml of water, boiled and centrifuged.

<table>
<tr><td colspan="2">Residue.Added hot water, decanted 10 drops of NH_4OH is added.Warmed and centrifuged.</td><td colspan="2">Centrifugate added drops of con. H_2SO_4 boiled. Carefully added 1 ml of water centrifuged.</td><td rowspan="2">Colors of the precipitates

1.Mercury White
Turning grey
2. Silver Yellow
3. Tungsten Blue
4. Lead Yellow
5. Thallium
Yellow</td></tr>
<tr><td rowspan="2">Residue Black

Added 3 drops of con. HCl,1 drop of con.HNO_3, boiled and centrifuged.

To the centrifugate added 3 drops of stannous chloride. White precipitate turning grey shows the presence of Mercury.</td><td colspan="2">Centrifugate Added dil.HCl in drops till a precipitate is formed. Added ammonium hydroxide and redissolved the precipitate. Added KI and centrifuged.</td><td rowspan="2">Residue White
Added 5 drops of NH_4OAc warmed. Added 2 drops of acetic acid and 2 drops of K_2CrO_4Yellow precipitate Show the presence of lead</td><td rowspan="2">Centrifugate Added NH_4OH and 2 drops of KI and sodium thiosulphate. Yellow precipitate Shows the presence of Thallium. Thallium is confirmed by flame test (Green Flame)</td></tr>
<tr><td>Residue Yellow Insoluble inNH_4OH</td><td>Centrifugate
Concentrate the solution and added two drops of stannous con. Chloride acid warmed. Blue precipitate shows the presence of tungsten</td></tr>
</table>

Mercury	**Silver**	**Tungsten**	**Lead**	**Thallium**	

ANALYSIS OF IA GROUP

<table>
<tr><td colspan="3">I A group precipitates is taken and added 2 drops of con.HCl and 2 drops of bromine water. Boiled, added 5 drops of saturated ammonium chloride and centrifuged.</td><td rowspan="4">Colour of precipitates.

1. Platinum Orange yellow
2. Gold Brown
3. Palladium Yellow
4. Selenium Red
5. Tellurium Blue</td></tr>
<tr><td rowspan="3">Residue Orange yellow presence of platinum</td><td colspan="2">Centrifugate Added 2 crystals of oxalic acid boiled and centrifuged.</td></tr>
<tr><td>Residue

Brown precipitate</td><td>Centrifugate Added NH_4OH in exceess dil HCl and centrifuged.</td></tr>
<tr><td>Residue Yellow crystals. Presence of palladium</td><td>Centrifugate Added a small quantity of hydroxylamine hydrochloride. Warmed and centrifuged.</td></tr>
</table>

Platinum	**Gold**	**Palladium**	**Residue**: Red precipitate Presence of Selenium **Selenium**	**Centrifugate** Added a crystal of hydrochloride boiled. Blue black crystalline precipitate. Presence of Tellurium. **Tellurium**	black

SEPARATION OF IIA AND IIB GROUP

The second group precipitate is taken and added with 1ml of NaOH. Boiled and centrifuged.

<table>
<tr><td colspan="2">Residue. IIA group precipitate</td><td colspan="2">Centrifugate. Added dil.HCl is added and neutralized. Boiled and centrifuged. The precipitate washed with water and analysed for IIBgroup.</td><td rowspan="5">Colour of the precipitates

1. Mercury Greyish white

2. Lead Yellow

3. Bismuth White Turbidity

4. Copper Reddish Brown

5. Cadmium Yellow</td></tr>
<tr><td colspan="4">Analysis of IIA group
The II A group precipitate is taken washed with water. To the residue 1.5 ml of dil.HNO_3 is added and boiled. Then
2drops of dil.H_2SO_4 added and centrifuged.</td></tr>
<tr><td colspan="2">Residue. Washed with 1ml of water and centrifuged. To the precipitate added NH_4OAc boiled and centrifuged.</td><td colspan="2">Centrifugate. Added NH_4OH in excess boiled and centrifuged.</td></tr>
<tr><td>Residue. Added 3 drops of con.HCl and one drop of con.HNO_3 boiled diluted with water. Added 2drops of stannous chloride. Greyish white precipitate.</td><td>Centrifugate. Added 1 drop of acetic acid and 2drops of K_2CrO_4. Yellow precipitate. Presence of lead.</td><td>Residue. Added dil.HCl and boiled to dissolve. This solution is added to excess of water taken in a beaker. White precipitate or turbidity shows Presence of Bismuth.</td><td>Centrifugate. Divided into two portions
1. To one portion added acetic acid and $K_4Fe(CN)_6$. Reddish brown precipitate confirms Copper.
2. To the second portion added con.HCl and excess water then passed H_2S gas. Yellow precipitate confirms Cadmium.</td></tr>
</table>

Mercury	**Lead**	**Bismuth**	**Copper and Cadmium**	

ANALYSIS OF IIB GROUP

The II B group precipitate is taken added NH_4Cl and few drops of con.HCl stirred and boiled. Diluted with water and centrifuged.

<table>
<tr>
<td colspan="2">Residue 5 drops of $(NH_4)_2CO_3$ is added, stirred and centrifuged.</td>
<td rowspan="2">Centrifugate. Divided into two portions.
1. To one portion Zn dust is added, boiled and dissolved. To this 3 drops of $HgCl_2$ is added. White or grey precipitate shows the presence of Tin.
2. To the second portion oxalic acid crystal is added and diluted with water, passed H_2S. Orange precipitate shows the presence of Antimony.

TIN AND ANTIMONY</td>
<td rowspan="2">Colour of the precipitates
1. Molybdenum Red alcohol layer
2. Arsenic Yellow
3. Tin White or grey
4. Antimony Orange</td>
</tr>
<tr>
<td>Residue Dark brown added. 3 drops of con.HCl and 2 drops of Br_2 water. Boiled to expel excess Br_2 diluted. Added 10% NH_4CNS, 3 drops of $SnCl_2$ and 10 drops of amyl alcohol. Shaken well.

Red alcohol layer shows the presence of Molybdenum.</td>
<td>Centrifugate. Acidified with dil.HCl.

Yellow precipitate shows the presence of Arsenic.</td>
</tr>
</table>

MOLYBDENUM	**ARSENIC**		

ANALYSIS OF III GROUP

To the III group precipitate added minimum amount of HCl and dissolved. Oxalic acid crystal is added. Digested in hot and centrifuged.

Residue. Added 1 ml $(NH_4)_2\ C_2O_4$. Boiled and centrifuged.	**Centrifugate:** NH_4OH is added and neutralized. Digested in hot & centrifuged. The residue is washed with NH_4Cl. Added 1ml of H_2O and 50 mg of Na_2O_2. Boiled till the effervescence stops centrifuged.

<table>
<tr>
<td>Residue. Added 3 drops of NaOH boiled& centrifuged. The residue is dissolved in dil.HNO_3. It is divided into 2 portions.

1. To one portion added NH_4OH and 6% H_2O_2. Boiled .Yellowish brown precipitate shows Cerium.

2. To the second portion added 2 drops of con.HNO_3 and boiled concentrated. A drop of 5% alcoholic solution of anthranilic acid is added. Dark blue precipitate dissolves rapidly and giving brown solution Shows the presence of Cerium</td>
<td>Centrifugate. 5 drops of dil.HCl is added. White precipitate added 5 drops of NaOH boiled and centrifuged. The residue is dissolved in dil.HCl and neutralized with NH4OH.
5 drops of m-nitrobenzoic acid is added &heated to 80°C white precipitate. Shows the presence of thorium .</td>
<td>Residue. Dissolved in dil.HCl and boiled. It is divided into 3 portion.
1.To one portion added 2 drops of KI and $Na_2S_2O_3$.
Yellow precipitate shows the presence of Thallium.

2.To the second portion, added H_3PO_4 to decolorize iron.2 drops of 6% H_2O_2 and 2 drops of dil H_2SO_4 is added.
White precipitate shows Zirconium.
Orange colour solution shows titanium. It is centrifuged.
<table>
<tr><td>Residue
White precipitate shows Zirconium</td><td>Centrifugate
Added 20mg of $Na_2S_2O_3$ boiled white precipitate shows Titanium</td></tr>
</table>
3.To the third portion a drop of con.H_2SO_4 is added concentrated. Added 5 drops of HNO_3 & 50mg of $NaBiO_3$. Stirred and allowed to stand.Purple colour of $KMnO_4$ shows presence of Manganese.</td>
<td colspan="4">Centrifugate:Acidified with dil.HNO_3. Added 5 drops $Pb(NO_3)_2$ and 200 mg of NH_4OAc crystals. Stirred well and centrifuged.
<table>
<tr>
<td>Residue .10 drops of dil.HNO_3 is added, boiled , dissolved and cooled. Added amyl alcohol and 6% H_2O_2 shaken.
(i)Blue alcohol layer shows Chromium
(ii) Reddish brown aqueous layer is divided into 2 parts .

1.To one part of 3 drops of dil.HCl is added boiled and cooled 2% aqueous solution (2 drops) of Tannin &NH_4OH is added.

2.To the second part NH_4OH is added and H_2S is passed .Red colour shows Vanadium</td>
<td colspan="3">Centrifugate.Added 3 drops HCl. Passed H_2S centrifuged. The precipitated Pbs is rejected. The centrifugate is boiled and cooled and $(NH_4)_2CO_3$ solution is added, boiled and centrifuged.
<table>
<tr><td colspan="2">Residue. Dissolved in dil.HCl added 2 drops of Na_2 S_2O_3 boiled &centrifuged.</td><td rowspan="2">Centrifugate Concentrated and added dil.HCl$K_4[Fe(CN)_6]NH_4OH$. Brown precipitate Few drops added NaOH. Yellow colour shows the presence of Uranium.</td></tr>
<tr><td>Residue
H_2O& few drops of $Co(NO_3)_2$ solutions added and shaken. A piece of filter paper is dipped and burnt.
Blue tinted ash (Thernard's blue) shows the presence of Aluminum.</td><td>Centrifugate.
Added quinalizarin. Blue colour shows the presence of Beryllium</td></tr>
</table>
</td>
</tr>
</table>
</td>
</tr>
<tr>
<td>Cerium</td>
<td>Thorium</td>
<td>Titanium</td>
<td>Vanadium</td>
<td>Aluminum</td>
<td>Beryllium</td>
<td>Uranium</td>
</tr>
</table>

Analysis of IV group

To the IV group precipitate 5 drops of HCl is added and shaken well and centrifuged.

Residue. Added 10 drops of conc.HCl and 1 crystal of $KClO_3$ concentrated and	**Centrifugate.** Boiled, A slight excess of NaOH is added and centrifuged	

divided into 2 portions. 1. To the portion, NH_4CNS & amyl alcohol is added and shaked well. Blue alcohol layer shows the presence of Cobalt 2. To the second portion,DMG and excess of NH_4OH is added. Scarlet precipitate shows the presence of Nickel. **Nickel**	**Residue** Turns brown in air. Add dil.HNO_3 50mg of Na BiO_3, stirred and centrifuged. Pink colour solution shows the presence of Manganese. **Manganese**	Centrifugate. Dividd into 2 portions 1. To one portion,H_2S gas is passed, Dirty white precipitate. 2. To the second portion HOAC and K_4 [Fe $(CN)_6$] is added. White precipitate shows the presence of Zinc. **Zinc**	Colour of the precipitates 1. **Cobalt** Blue alcohol layer 2. **Nickel** Scarlet precipitate 3. **Manganese** Pink colour 4. **Zinc** White precipitate

ANALYSIS OF V GROUP

The precipitate from V group is dissolved in minimum portion ofdil.HOAc.The solution is divided into three portions.

1. To the first portion 3 drops of K_2CrO_4 is added yellow precipitate shows Barium. 2. Flame test is performed from the original mixture. Green flame shows Barium **Barium**	1. To the second portion added six drops of ammonium sulphate. White precipitate, confirms Strontium. 2. Flame test is performed with the original mixture crimson red confirms Strontium **Strontium**	1. To the third portion added NH_4OH and 5 drops of ammonium oxalate white precipitate confirms Calcium. 2. Flame test is performed with original mixture. Brick red colour confirms Calcium. **Calcium**	Colours of the flame 1. **Barium** Green 2. **Strontium** Crimson red 3. **Calcium** Brick red

Analysis of VI group

Experiment	Observation	Inference
1. To the original mixture taken in a test tube added NH_4OH and warmed.	Colourless gas with pungent smell giving dense white fumes with a glass rod dipped in NH_4OH.	Presence of ammonium
2. To the original solution added a small quantity of Nessler's reagent.	A Brown precipitate	Presence of ammonium
3. To the original solution added NH_4Cl, NH_4OH and Na_2HPO_4. The inner side of the test tube is scratched with a glass rod.	White precipitate	Presence of Magnesium and Lithium
4. Flame test is performed with original mixture.	Caramine red colour	Presence of Lithium is confirmed.

Result: The given mixture contains …… and …… as common cations and
…… and …… as less common cations.

SPOT TEST

1. Thallium

To one drop of the original solution , one drop of KI and two drops of sodium thiosulphate solution is added. An yellow or brownish black shows Thallium.

2. Tungsten

To a little of salt solution added Ferrous sulphate solution. A brown precipitate is formed. To this precipitate dil.HCl is added and heated. The precipitate turns white and then yellow.

3. Selenium

[a] To the original solution of the mixture added KCN and 2 ml of Con.HCl and boiled. A red precipitate is formed.

[b] To a small quantity of the original solution added Thio urea solution orange red precipitate shows the presence of Selenium.

4. Molybdenum

[a] To a small quantity of the original solution added one or two drops of KCN solution. A reddish brown precipitate soluble in NaOH gives the presence of Molybdenum.

[b] A drop of the test solution is mixed with the drop of Phenyl hydroxime hydrochloride solution. A red colourisation solution is produced presence of molybdenum.

[c] To a small quantity of the original solution added ferrous sulphate solution upon added dil. HCl the colour changes to blue and then green on warming but and then returns to blue on cooling

(Difference from tungsten)

5. Titanium

To a little original solution added a small quantity of Zinc powder. A Violet colourisation is produced presence of Titanium.

6. Thorium

To a small quantity original solution added for two drops of Potassium ferrocyanide, a white precipitate shows the presence of Thorium.

REACTIONS OF THE III GROUP LESS COMMON IONS

S.NO	Test	Titanium	Zirconium	Cerium	Thorium	Beryllium	Vanadium	Uranium
1	NH_4OH	White gelatinous precipitate	White gelatinous precipitate	Yellow precipitate	White precipitate	White precipitate	-	Yellow precipitate dissolving in $(NH_4)_2 CO_3$.
2	H_2O_2	Orange yellow colouration in presence of 2 drops of dil.H_2SO_4	-	Yellow brown precipitate in the presence of NH_4OH	White precipitate	-	A red coloration is produced	Pale yellow precipitate
3	NaOH	-	A White gelatinous precipitate	Yellow precipitate	White precipitate	-	-	Yellow precipitate soluble in $(NH_4)_2 CO_3$.
4	Oxalic acid	-	White precipitate	White precipitate	White precipitate	-	-	-
5	Ammonium sulphide	-	White gelatinous precipitate	White precipitate	White precipitate	-	Red colour to brown	Brown precipitate

6	Silver Nitrate	-	-	Black precipitate	-	-	-	-
7	Potassium iodide	-	-	White precipitate	White precipitate	-	-	-
8	Ammonium carbonate	-	-	White precipitate	White precipitate	White precipitate	-	White precipitate

INORGANIC PREPARATIONS

Preparation of Tetrammine Copper(II) Sulphate

Chemicals Required

Copper sulphate penta hydrate - 5g

Concentrated ammonia - 7.5ml

Ethanol - 10ml

Procedure:

Dissolved 5g of copper sulphate penta hydrate in 10ml of deionised water. Added 7.5ml of concentrated 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, washed with 1:1 alcohol-ammonia solution and dried. The complex is recrystallized from 1:1 alcohol-water mixture.

$CuSO_4.5H_2O+4NH_3 \rightarrow [Cu(NH_3)_4]SO_4+5H_2O$

Result:

Yield of the product = ---------- g

Preparation 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 portions, 1g of potassium oxalate in the solution. Heated the solution to boiling and dissolved 1.3g potassium oxalate in that solution. The cooling is continued until the blue green crystals separated. The sample is recrystallized by using 1:1 water alcohol mixture.

Result:

Yield of the product = ---------- g .

Preparation of Potassium Trioxalato Ferrate (III)

Chemicals Required

Ferrous oxalate - 5g

Potassium oxalate -5g

Hydrogen peroxide -15ml

Oxalic acid -15ml

Ethanol -10ml

Procedure

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

Result

Yield of the product = ----------

Preparation of Copper (I) Chloride

Chemicals Required

Copper sulphate pentahydrate - 10g

Sodium chloride - 5g

Copper turnings - 10g

Hydrochloric acid(Concentrated) - 30ml

Na_2SO_4, Alcohol, Ether - 4g

Procedure

10 gram of copper sulphate pentahydrate, 5 g of sodium chloride and 10 g of copper turnings are mixed intimately and then placed in a 250 ml conical flask about 30 ml of con.HCl is added to the flask and the mixture is heated in a water bath for nearly one hour until the blue or bluish green colour disappears completely. When the solution in the flask acquires a straw colour, it is poured in a beaker containing about 500 ml of water containing 3 to 4 gof $Na_2SO_4.10H_2O$. A dull white precipitate of cuprous 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 desiccators.

Result

Yield of the product = ---------- g.

Preparation of Hexammine Cobalt (III) Chloride

Chemicals Required

Ammonium Chloride - 12g

Cobaltous chloride -18g

20 % H_2O_2 -35ml

Procedure

Cobalt chloride is dissolved in the boiling solution of 12 gram of Ammonium Chloride in 25 ml of water and 1 g of animal charcoal and cool the contents in the running water. Wash out the vessel in which the solution was first made with 40 ml of con.NH_3 and then ammonical liquid to the flask cool the whole contents of preparation in ice bath to 10ºC. 35 ml of 20 % H_2 O_2 is added slowly in portion while briskly shaking the flask and its contents. All the oxidizing agents have been added heat mixture gradually 60 º C with mixing by shaking until the pinkish tint in the liquid removed crystals being separate at the close of heating and deposit in quantity on cooling in on ice bath. Filter with the crude solid and without washing it, transfer it to a beaker containing a boiling of a mixture of 150 ml of water and 5 ml of con.HCl when all solid accept the charcoal is dissolved filter in liquid

while hot, add 20 ml of 10 con.HCl to the filtrate and then cool the liquid in an ice bath. Golden brown crystal separate out.

Result

Yield , the amount of hexamine cobalt (III) chloride = ---------- g.

Preparation of Ammonium hexachloratestannate (IV)

Chemicals Required

Tin -10g

Ammonium Chloride -9g

Concentrated HNO_3

Conc.HCl

Procedure

10g of the tin is placed in a flask and warmed with conc.HCl until the metal begins to dissolved displaying hydrogen then add conc.HNO_3 in sufficient quantity to oxidize the Stannous (II) to stannic (IV) state. When the metal has dissolve, test a few drops of the solution with $HgCl_2$. If a white precipitate is formed, stannous tin is present and a little more concentrated nitric acid must be added to oxidize it . Filter the stannic solution.

9g ammonium chloride is dissolved in a small quantity of hot water mix these two solutions and evaporate until on cooling ammonium hexachlorostannate (IV) crystallizes out from the solution. Filter at the pump, wash with a little conc.HCl acid and dry it.

Result

The amount of Ammonium HexachloroStannate (IV) = ---------- g.

Preparation of ortho and para hydroxyl phenol mercuric chloride

Chemicals Required

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

Procedure:

3g of phenol is melted in a beaker of 150ml capacity by setting the beaker with contents 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 briskly stir the mixture. Boil the contents of the beaker over a small free flame and if necessary add continuously a small amount of boiling water until on vigorous shaking the oil dissolves leaving only a small residue. This residue usually becomes firmly attached to the walls of the beaker and the clear solution can readily be decanted from it.

On dissolving 1.25g of the sodium chloride in the hot solution, its p-isomer at once separates and is rendered granular by keeping it on the boiling water bath for 10minutes. After filtering p-isomer from the hot liquid, the other isomer crystallises from the cooling filtrate. The p-isomer is washed on the filter with hot water and more soluble ortho isomer with a small quantity of cold water.

Result:

Amount of the two isomers obtained

o – Compound = ---------- g.
p- Compound = ---------- g.

Preparation of Tris[Thio-urea]Copper(I) Chloride

Chemicals Required

Cupric chloride dehydrate - 2.7g

Thio urea - 5g

Concentrated hydrochloric acid - 5ml

Procedure:

Dissolved 5g of thiourea in 30ml of hot water. To the solution added 2.7g of cupric chloride dehydrate and 5ml concentrated hydrochloric acid in 15ml of deionised water. Heat on a boiling water bath. Filtered the hot solution and allowed the clear solution to cool. Filtered the product recrystallized the sample from 5% aqueous thiourea solution or with small amount of dilute hydrochloric acid

Result:

Yield of the product = ---------- g.

Preparation of Chrome Alum

[$K_2SO_4.Cr_2(SO_4)_3.24H_2O$]

Chemicals Required

Concentrated sulphuric acid - 12.5ml

Potassium dichromate - 15g

Alcohol - 7ml

Procedure:

In a 400ml beaker diluted sulphuric acid (12.5ml of con.H_2SO_4 is 11.5ml of water) is taken and 15g of $K_2Cr_2O_7$ powder is added. It is cooled in a ice cold water bath and added 7ml of alcohol with constant stirring. The temperature should be maintained at or near 50^oC (if necessary dropped of a piece of a ice in the mixture). The solution is concentrated in a water bath below 60^oC. The solution is cooled in a ice bath. The contents are allowed to stand overnight and the crystals are collected. The product obtained is recrystallized from cold water.

Result:

Yield of the product = ---------- g.

Preparation of Potassium Trioxalato Aluminato (III)

Chemicals Required

Aluminum powder - 0.5g

Potassium hydroxide - 20g in 100ml

Oxalic acid - 7g

Procedure

Weighed about 0.5 g of commercial aluminum powder into 400 ml beaker. Added 15 ml of solution of 20% potassium hydroxide. Heated to boiling to dissolve aluminum. Filtered the solution and added 10 ml of water to the filtrate and the solution is heated to boiling. Weighed about 7 g of oxalic acid and added to the boiling solution. The solution filtered and cooled.When the solution gets the room temperature added about 10 ml 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

Yield of the product = ---------- g.