[1] Determination of Fe by gravimetrically and Cu by volumetrically.

Separation of Cu and Fe

Take the 50ml solution from O.S.(250ml), heat the solution add 5ml 2N HCl and pass the H_2S. Black ppt's of CuS is obtained, filter the CuS ppt's, wash the ppt's using dill water (or H_2S water) and collect the filtrate and washing carefully in beaker. Remove the unused H_2S from solution by boiling the solution. Use this solution for estimating of Fe by gravimetrically. Remove the ppt's of CuS

Gravimetric estimation of Fe [Fe_2O_3].

Take the solution of Al in 500ml beaker. To this solution add 50ml distilled water and then add 2-3gm of NH4Cl and 1-2 drops of Methyl red. Heat the solution to boiling. Now discontinue heating. To this solution add 50%. NH4OH solution drop wise with stirring till the colour of solution is becomes yellow. Boil the solution again 1-2 minutes and allow the ppts to settle down. Test the supernant liquid for complete precipitation. Filter the ppts on What-man filter paper No-1. Wash the ppt. using 1% NH_4NO_3 or D.W. until the washing free from Cl and SO_4 ions. Allow the filter paper to drain thoroughly. Dry the ppt on a hot air cone. After drying, remove the filter paper from the funnel and fold the edges over ppts to packet and put into a weighed crucible. Heat the ppts for half an hour. Cool the ppts and weight the ppts of **Fe_2O_3**.

Volumetric estimation of Cu.

Pipette out 25ml of original solution into a conical flask from above solution. Neutralized solution using dil. Ammonia solution and acetic acid solution. To this add 1 TT of 10% KI solution and add 5ml of starch. Titrate with 0.01M $Na_2S_2O_3$ until the solution becomes white colour. Note down end point.

[2] Determination of Ni by gravimetrically and Zn by volumetrically.

Gravimetric estimation of Ni [$Ni(DMG)_2$].

Take the 50ml solution from O.S.(250ml) in 500ml beaker. To this solution add 50ml distilled water and 1-2 drops of Methyl red. Then add 25-30ml of 1% DMG with constant stirring. Now go on adding 1:1 NH4OH solution drop wise till the solution become alkaline (Yellow colour). Digest the ppt's on water bath for about 20 minutes. Then test for complete precipitation by adding few drop of DMG. Filtrate the ppts in filter paper, which are equal weighed. Wash the ppts with hot water. Collect the filtrate and washing in clean beaker and use the filtrate for estimating of Zn. Now dry the ppts at 110 C in an electric oven for about an hour. Cool the ppts and weight.

Volumetric estimation of Zn.

Transfer the filtrate and washing obtain from above step to beaker and evaporate it to 80 to 100ml. Filter the solution and add 5ml of Con HNO_3 and 3ml of con. HCl and evaporate again. Now add 1ml con. H_2SO_4 and evaporate till thick white fumes of sulphur trioxide appear. Cool the solution and take the solution in the 250ml measuring flask and make up the volume up to the mark with D.W.

Pipette out 25ml solution from above into a conical flask. To this add 25ml d.w.,and Add 5ml of 10pH buffer solution and 2-3 drops of EBT indicator. Titrate with 0.01M EDTA until the colour changes from wine red to Blue.

[3] Determination of Ni by gravimetrically and Cu by volumetrically.

Separation of Cu and Ni

Take the 50ml solution from O.S.(250ml), heat the solution add 5ml 2NHCl and pass the H_2S. Black ppt's of CuS is obtained, filter the CuS ppt's, wash the ppt's using distilled water (or H_2S water) and collect the filtrate and washing carefully in beaker. Remove the unused H_2S from solution by boiling the solution. Use this solution for estimating of Ni by gravimetrically.

Collect the ppt's of CuS in beaker and boil with 50% of HNO_3 with constant starring until all ppts are soluble. Add 1ml of con HCl and boil the solution for removing excess NO2 from solution. Make the solution 100ml in measuring flask. Use this solution for estimating of Cu by Complexometric titration using EDTA.

Gravimetric estimation of Ni [$Ni(DMG)_2$].

Take the solution of Ni in 500ml beaker. To this solution add 50ml distilled water and 1-2 drops of Methyl red. Then add >30ml of 1% DMG with constant stirring. Now go on adding 1:1 NH_4OH solution drop wise till the solution become alkaline (Yellow colour). Digest the ppt's on water bath for about 20 minutes. Then test for complete precipitation by adding few drops of DMG. Filtrate the ppts in filter paper, which are equal weighed. Wash the ppts with hot water. Now dry the ppts at 110 C in an electric oven for about an hour. Cool the ppts and weight.

Volumetric estimation of Cu.

Pipette out 25ml of Cu solution into a conical flask from above solution. To this add 25ml d.w.,5ml of con. Ammonia solution and 2-5 drops of the Fast Sulfone Black-F indicator. Titrate with 0.01M EDTA until the colour changes from Blue (Violet) to a deep green.

<u>Calculation of amount of Ni.</u>

Observation: Weight of Complex = _____ gm.

Ni[C8H14N4O4] = Ni
288.71 gm of complex = 58.7 gm Ni

_____gm complex= $\frac{\text{_____}\times 58.7}{288.71}$ = _____ gm Ni in 50ml of Solution.

Amount of Ni in given 250 ml Solution = = $\frac{\text{_____}\times 250}{50}$ = _____gm Ni in 250ml Solution.

<u>Calculation of amount of Cu.</u>

Observation: Volume of 0.01 M EDTA = _____ ml.

1000 ml 1M EDTA = 63.5 Cu^{+2}

_____ml 0.01 M EDTA = $\frac{\text{_____}\times 63.5\times 0.01}{1000}$ = _____gm Cu^{+2}in 25 ml solution.

Amount of Cu in 100 ml solution = $\frac{\text{_____}\times 100}{25}$ = _____ gm Cu^{+2} in 100ml solu.

Amount of Cu in given 250 ml solution== $\frac{\text{_____}\times 250}{100}$ _____ gm Cu^{+2} in 250ml solution.

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

Aim : To prepare Hexamine Nickel(Ⅱ)chloride $[Ni(NH_3)_6]Cl_2$, and determine the % purity of complex.

Requirements: Nickel chloride=12 gm; Liq. Ammonia=>50 ml; Acetone=3Test tubes

Method:

Dissolve 12 gm of Nickel chloride in minimum quantity of warm water. Add about 25ml liq. Ammonia slowly to the rapidly stirred solution until the green colour Nickel hydroxide precipitate has dissolved and obtained dark blue colour of solution. Allow the solution for 30 minutes at room temp. Then add 3 T.T of acetone. Purple color precipitate are obtaining. Filter the ppts & dry it. Wash ppt. using ammonia and acetone.

Calculation:

1. Theoretical yield of complex.
   $NiCl2.6H2O$ = $[Ni(NH_3)_6]Cl_2$
   237.7gm of $NiCl2.6H2O$ = 231.7 gm of $[Ni(NH_3)_6]Cl_2$

$$= \frac{12 \times 231.7}{237.7} = 11.7\ gm\ complex$$

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times \_\_\_\_}{11.7} = \_\_\_\_\_\%$$

Estimation of Ni(II) using Muroxide as indicator(direct titration)

Prepare a complex solution

Weigh out exactly 0.500 to 1.000 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 – 3 ml of Conc. HCl. Then evaporate the sol^n to remove excess $NO_{2(gas)}$. Make the sol^n 250 ml

Volumetric estimation of Ni

Pipette out 25ml of nickel ion solution into a conical flask. 5-6 drops of muroxide indicator and 5ml of 2M ammonium chloride solution. Now add con. Ammonium solution (5-8ml) drop wise until the pH of the solution becomes 7 which is shown by the yellow colour of the solution. Titrate with 0.01M EDTA until the colour changes from yellow to blush violet.

1.Theoretical amount of Ni^{+2} in complex.

$[Ni(NH_3)_6]Cl_2$ = Ni^{+2}

231.7 gm complex = 58.71 gm Ni^{+2}

_____ gm complex $= \frac{\_\_\_\_\_ \times 58.71}{231.7}$

= _____ gm Ni^{+2} in complex.

2.Practical amount of Ni^{+2} in complex.

1000 ml 1M EDTA = 58.71 gm Ni^{+2}

_____ ml 0.01 M EDTA $= \frac{\_\_\_\_\_ \times 58.71 \times 0.01}{1000}$ = _____ gm Ni in 25 ml of complex solution.

In 250 ml complex solution (_____ gm complex), amount of Ni $= \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Ni^{+2} in complex.

3.% of purity of complex. $= \frac{PA \times 100}{TA}$

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

Aim : To prepare tetramine cupric sulphate and determine % of purity.

REAGENTS :Cupric sulphate, Ammonia, Ethyl alcohol, Distilled water, Sulphuric acid.

REACTION: $CuSO_4 \cdot 5H_2O(aq) + 4NH_3(aq) = [Cu(NH_3)_4]SO_4 \cdot H_2O + 4H_2O$ (C)

PROCEDURE : Take 3 gm crystalline cupric sulphate in a 250 ml beaker. Dissolve it in minimum quantity of water and then add few drops of diluted sulphuric acid. Add concentrated ammonia solution to the beaker with constant stirring, until the blue precipitate of cupric hydroxide, first formed completely dissolve to yield a clear, deep blue solution and there should be smell of ammonia in the beaker. Now add 20 ml alcohol dropwise from the dropping funnel to the beaker with constant stirring until the blue precipitates settled and clear solution is obtained. Tetramine cupric sulphate separates out. Filter and wash the crystals with a few drops of alcohol. Dry the crystals and weigh the dry crystal and find out the percentage yield followed by percentage purity by usual methods.

Calculation:

1. Theoretical yield of complex.
   $CuSO_4 \cdot 5H_2O = [Cu(NH_3)_4]SO_4 \cdot H_2O$
   159.61 gm of TACS = 245.79 gm of complex
   $$= \frac{3 \times 245.79}{159.61} = 4.620 gm\ complex$$
2. Practical Yield : _____ gm complex.
3. % of Yield
   $$= \frac{100 \times \_\_\_\_}{4.620} = \_\_\_\_\_\%$$

Prepare a Cu^{+2} complex solution:

Weigh out exactly 0.500 to 1.000 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 – 5 ml of Conc. HCl. Then evaporate the sol^n to remove excess $NO_{2(gas)}$. Make the sol^n 250 ml.

Estimation of Cu Volumetrically (% purity of complex)

Take 25 ml of stock solution in conical flask. Add 10 ml ammonia to make it alkaline and add few drops of Fast Sulphone Black-F indicator. Titrate it against 0.01 M EDTA solution, until the color changes from blue to dark green. Repeat it until constant reading. Tabulate your results.

1.Theoretical amount of Cu^{+2} in complex.

$[Cu(NH_3)_4]SO_4 \cdot H_2O = Cu^{+2}$

327 gm complex = 63.5 gm Cu

_____ gm complex $= \frac{\_\_\_\_\_ \times 63.5}{327}$ = _____ gm Cu^{+2} in complex.

2.Practical amount of Cu^{+2} in complex.

1000 ml 1M EDTA = 63.5 Cu^{+2}

_____ml 0.01 M EDTA $= \frac{\_\_\_\_\_ \times 63.5 \times 0.01}{1000}$ = _____ gm Cu^{+2} in 25 ml of complex solution.

In 250 ml complex solution (_____ gm complex), amount of $Cu^{+2} = \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Cu^{+2} in complex.

3.% of purity of complex. $= \frac{PA \times 100}{TA}$

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

Aim :To Prepare Ammonium Tetrathiocyanatodiammine chromate $NH_4[Cr(NH_3)_2(CNS)_4]$, and Determine % of purity of complex.

Requirements: Ammonium thiocynate (NH_4CNS) = 8.0 gm.; Ammonium dichromate (NH_4CrO_7) = 3.0 gm.; Ethyl Alcohol and cold Water

Equation: $\boldsymbol{8NH_4CNS + (NH_4)_2Cr_2O_7 \rightarrow 2NH_4[Cr(NH_3)_2(CNS)_4] + 4NH_3 + 7H_2O}$

Method:

Take 8 gm Ammonium thiocynate in silica dish and heat it slowly. When it start for melting add 3 gm Ammonium dichromate slowly time to time with continues stirring. Evaporate excess ammoniya, Violet color solid part is obtained. Then cool at room temperature. Then add some ice cold water in it for washing excess amount of mother solt. Orange colored ppts obtain. Wash the ppts & fiter it & Dry it.

Calculation:

1. Theoretical yield of complex.
   $\boldsymbol{(NH_4)_2Cr_2O_7}$ = $\boldsymbol{2NH_4[Cr(NH_3)_2(CNS)_4]}$
   252.07 gm of ADC = 682 gm of complex
   $$= \frac{3 \times 682}{252.07} = 8.116\ gm\ complex$$
2. Practical Yield : _____ gm complex.
3. % of Yield
   $$= \frac{100 \times \_\_\_\_}{8.12} = \_\_\_\_\_\%$$

Estimation of Cr^{+3}

Prepare a complex solution:

Weigh out exactly 0.500 to 1.000 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 – 5 ml of Conc. HCl. Then evaporate the sol^n to remove excess $NO_{2(gas)}$. Make the sol^n 250 ml.

Volumetric estimation of Cr(III)

Take 25 ml diluted solution in a conical flask add 25 ml 0.01 M EDTA solution, then add Hexamine buffer to adjust the pH 5 to 6. Add Xylenol Orange indicator, excess of EDTA is titrate against 0.1 M Pb (NO3)2 solutions. Color change brownish orange to red. Repeat it until constant reading. Tabulate your results. Find out the yield and percentage purity. Find out the yield and percentage purity. (Here 25 ml - Burrett reading = Bml)

1.Theoretical amount of Cr^{+3} in complex.

$\boldsymbol{NH_4[Cr(NH_3)_2(CNS)_4]}$ = Cr^{+3}

341 gm complex = 52.0 gm Cr^{+3}

_____ gm complex $= \frac{\_\_\_\_\_ \times 52.0}{341}$ = _____ gm Ni^{+2} in complex.

2.Practical amount of Cr^{+3} in complex.

1000 ml 1M EDTA = 52.0 Cr^{+3}

(25-BR)ml 0.01 M EDTA $= \frac{\_\_\_\_\_ \times 52.0 \times 0.01}{1000}$ = _____ gm Cr^{+3}in 25 ml of complex solution.

In 250 ml complex solution (_____ gm complex), amount of $Cr^{+3} = \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Cr^{+3} in complex.

3.% of purity of complex. $= \frac{\boldsymbol{PA \times 100}}{\boldsymbol{TA}}$

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

AIM: To prepare Tris(acetylactone)manganese (II) chelate [$Mn(CH_3COCHCOCH_3)_3$] & determine % of purity of complex.

Requirements: $KMnO_4$: 2.0 gm. Acetyl acetone : 10.0 ml.

Equation: 2KMnO4 + 6CH3-CO-CH2-CO-CH3 + 6 NaOH → 2Mn(acac)3 + 2K2CO3 + 3H2O

Method: Take 2 gm $KMnO_4$ in dry beaker, add 30 ml D.W. and 5ml of 2N NaOH, boil the soln. about 10-15 mini. Then cool it at room temperature. Then add 10 ml acetyl acetone drop by drop for continues starring. Boil again soln. for 5 min. Then cool the solution in ice bath. Collect the shiny brown-black crystals on a course fritted filter and wash them tree times with 10-mL portions of distilled water. Dry the crystals.

Calculation:

1. Theoretical yield of complex.
   $KMnO_4$ = **[$Mn(CH_3COCHCOCH_3)_3$]**
   158.03 gm of $KMnO_4$ = 352.26 gm of complex
   $$= \frac{2 \times 352.26}{158.03} = 4.455\ gm\ complex$$
2. Practical Yield : _____ gm complex.
3. % of Yield
   $$= \frac{100 \times \_\_\_\_}{4.45} = \_\_\_\_\_\%$$

Estimation of Mn(II):

Prepare a Mn^{+2} solution :

Weigh out exactly. 0.500 to 1.00 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 – 3 ml of Conc. HCl. Add some dil.HCl for transfer the sol^n Then heat till yellow color obtain. Then make it 250 ml.

Procedure: Take 25ml of Mn(II) solution in conical flask. Add 0.5gm of Hydroxylammonium chloride and 3ml of triethanolamine shake well solution. Add 4-5ml of 10 pH buffer solution and 2 to 5 drops of [EBT]Solochrome Black indicator. Titrate against 0.01 M EDTA solution until colour of solution become red to blue-green. Decide to end point.

OR

25 ml diluted Complex Sol^n + 0.5gm NH4Cl OR Ascorbic acid+ 4-5 ml of 10pH buffer solution + 2-4 drops of EBT. Titrate against 0.01 M EDTA solution until colour of solution become red to blue-violet. Decide to end point.

1.Theoretical amount of Mn in complex.

[$Mn(CH_3COCHCOCH_3)_3$] = Mn

352.26 gm complex = 54.94 gm Mn

_____ gm complex $= \frac{\_\_\_\_\_ \times 54.94}{352.26}$ = _____ gm Mn in complex.

2.Practical amount of Mn in complex.

1000 ml 1M EDTA = 54.94 Mn

_____ ml 0.01 M EDTA $= \frac{\_\_\_\_\_ \times 54.94 \times 0.01}{1000}$ = _____ gm Mn in 25 ml of complex solution.

In 250 ml complex solution (_____ gm complex), amount of Mn $= \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Mn in complex.

3.% of purity of complex. $= \frac{PA \times 100}{TA}$

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

Aim : To prepare cis-Potaassiumdioxalatodiaquochromate (III) $K_3[Cr(C_2O_4)_2(H20)_2]$ and determine the % of purity of complex.

Requirements: Potassium dichromate=3gm; Oxalic acid=6gm Ethanol.

Equation: $\boldsymbol{7C_2H_2O_4 + K_2Cr_2O_7 \rightarrow 2K[Cr(C_2O_4)_2(H_2O)2] + 6CO_2 + 13H_2O}$

Method: Take 3 gm of potassium dichromate and 6 gm of Oxalic acid fine crystals. Mix two powder and put mixture at the center of China dish in a compact heap and moisten it with water. Heat the China dish gently on a low flame, after a few minutes a vigorous reaction will set due to evaporation of carbon monoxide and water vapor. Without waiting for the thick liquid to cool and pour using ethanol with constant suturing. When complete solidification filtrate mixture and dry the ppt's and weight ppt's.

Calculation:

1. Theoretical yield of complex.
   $\boldsymbol{(K)_2Cr_2O_7}$ = $\boldsymbol{2K[Cr(C2O4)_2(H2O)_2]}$
   294.18 gm of PDC = 678.38 gm of complex
   $$= \frac{3 \times 678.38}{294.18} = 6.918\ gm\ complex$$
2. Practical Yield : _____ gm complex.
3. % of Yield
   $$= \frac{100 \times \_\_\_\_}{6.918} = \_\_\_\_\_\%$$

Estimation of Cr(III)

Prepare a Cr^{+3} solution :

Weigh out exactly 0.500 to 1.000 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 – 5 ml of Conc. HCl. Then evaporate the sol^n to remove excess $NO_{2(gas)}$. Make the sol^n 250 ml.

Volumetric estimation of Cr(III)

Take 25ml of chromium complex solution in conical flask and add 25 ml of distilled water. Add 25 ml of 0.01M EDTA and add 5 ml of 2M HNO3 (pH=1 to 2) boil the solution for 10 minutes when the violet colour of solution is produced. Now, add Hexamine power to bring pH 5 to 6 and Xylenol orange solution as an indicator and then titrate the excess of EDTA with 0.01M Pb(NO3)2 kept in burette. Define the end point.

1.Theoretical amount of Cr^{+3} in complex.

$\boldsymbol{K[Cr(C2O4)_2(H2O)_2]}$ = Cr^{+3}

339.19 gm complex = 52.0 gm Cr^{+3}

_____ gm complex $= \frac{\_\_\_\_\_ \times 52.0}{339.19}$ = _____ gm Cr^{+3} in complex.

2.Practical amount of Cr^{+3} in complex.

1000 ml 1M EDTA = 52.0 Cr^{+3}

(25-BR)ml 0.01 M EDTA $= \frac{\_\_\_\_\_ \times 52.0 \times 0.01}{1000}$ = _____ gm Cr^{+3}in 25 ml of complex solution.

In 250 ml complex solution (_____ gm complex), amount of $Cr^{+3} = \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Cr^{+3} in complex.

3.% of purity of complex. $= \frac{\boldsymbol{PA \times 100}}{\boldsymbol{TA}}$

M.Sc. Semester: 2 Preparation and Determination of Purity of Complex

Aim : To prepare Nickel dimethylglyoxime.

REAGENTS : $Ni(NH_4)_2(SO_4)_2.6H_2O$, Ammonia, 1% DMG.

PROCEDURE : Take 0.3 to 0.4 gm of Nickel ammonium sulphate and make clean solution using 20ml DW. To this solution add 10ml distilled water and 1-2 drops of Methyl red. Then add 25-30ml of 1% DMG with constant stirring. Now go on adding 1:1 NH_4OH solution drop wise till the solution become alkaline (Yellow color). Digest the ppt's on water bath for about 20 minutes. Then test for complete precipitation by adding few drops of ammonia. Filtrate the ppts in filter paper. Wash the ppts with hot water. Dry it into Owen at 100 C.

Calculation:

1. Theoretical yield of complex.
   $Ni(NH_4)_2(SO_4)_2.6H_2O$ = $Ni[C_8H_{14}N_4O_4]$
   286.9 gm of NAS = 288.71 gm of complex

$$= \frac{0.3 \times 288.71}{286.9} = 0.302\ gm\ complex$$

2. Practical Yield : _____ gm complex.
3. % of Yield

$$= \frac{100 \times \_\_\_\_}{0.302} = \_\_\_\_\_\%$$

Prepare a Ni^{+2} complex solution:

Weigh out exactly 0.100 to 0.300 gm of dry complex. Add minimum quantity of Conc. HNO_3 to dissolve it then add 1 –2 ml of Conc. HCl. Then evaporate the sol^n to remove excess $NO_{2(gas)}$. Make the sol^n 250 ml.

Estimation of Ni Volumetrically (% purity of complex)

Take 25 ml of diluted solution in conical flask. Add 4-5 ml of 10pH buffer solution. Add few drops of Murexide indicator. Titrate it against 0.01 M EDTA solution, until the color changes from yellow to violet. Repeat it until constant reading. Tabulate your results. Find out the yield and percentage purity.

1.Theoretical amount of Ni^2 in complex.

$Ni[C_8H_{14}N_4O_4]$ = Ni^{+2}

288.71 gm complex = 58.7 gm Ni

$$\_\_\_\_\_ \text{ gm complex} = \frac{\_\_\_\_\_ \times 58.7}{288.71} = \_\_\_\_\_ \text{ gm } Ni^{+2} \text{ in complex.}$$

2.Practical amount of Ni^{+2} in complex.

1000 ml 1M EDTA = 58.7 Ni^{+2}

$$\_\_\_\_\_ \text{ml 0.01 M EDTA} = \frac{\_\_\_\_\_ \times 58.7 \times 0.01}{1000} = \_\_\_\_\_ \text{ gm } Ni^{+2} \text{ in 25 ml of complex solution.}$$

In 250 ml complex solution (_____ gm complex), amount of Ni^2 $= \frac{\_\_\_\_\_ \times 250}{25}$

= _____ gm Ni^{+2} in complex.

3.% of purity of complex. $= \frac{PA \times 100}{TA}$