**Experiment No.1:**

**Preparation of tetraamine copper(II) sulfatemonohydrate, [Cu(NH3)4]SO4.H2O**



Cu: [Ar]3d104S1

Cu2+: 3d94S0





Square planar, paramagnetic

|  |  |
| --- | --- |
| CuSO4 | [Cu(NH3)4]SO4.1H2O |
| M.wt | M.wt |
| 1g | T=theoretical weight |

Yield%= (observed weight/ theoretical weight)×100

Error%= {(theoretical weight- observed weight)/ theoretical weight}×100

Experimental:

Dissolve 1g of CuSO4 in 3 ml water. Add ammonia solution (10 ml) then stir for 5 min. Add EtOH (5 ml) then stir for 15 min. Cool it in ice bath until the precipitate has been formed. Filter and dry in air.

[Cu(NH3)4]SO4.1H2O=245.75

CuSO4=159.6

**Experiment No.3:**

**Determination of ammonia in the complex**

**[Cu(NH3)4]SO4.H2O:**

[Cu(NH3)4]SO4.1H2OCu2+ +HCl+ NH4++.... .



V2

V1-V2=V3 volume of HCl reacted with NH3

1L 1M HCl Ξ 1L 1M NH3

1L 1M HCl Ξ 1mol NH3

1L 1M HCl Ξ 1M.wt NH3

1ml 0.1N HCl Ξ 1/10000 M.wt NH3

V3 ml 0.1N HCl Ξ (V3/10000) x 17 Ξ y wt of NH3 in 0.1g of the complex

Observed weight percentage (O) =(y x 100)/0.1

|  |  |
| --- | --- |
| [Cu(NH3)4]SO4.1H2O | NH3 |
| M.wt | 4M.wt |
| 0.1g | Z |

Theortical weight percentage (T) =(Z x 100)/0.1

Theortical weight percentage (T) =27%

Find Error%

Dissolve the complex (0.1g) in 35ml of HCl (0.1M).Titrate with NaOH solution (0.1M) using methylred as indicator.At the end point, the colour changes from light red to light orange.

Required materials:

1-HCl (0.1 M, 250 ml)

2-NaOH (0.1 M, 250 ml)

3-Methylred indicator

**Experiment No.5: Preparation of tris(acetylacetonato)iron(III)  
[Fe(C5H7O2)3]:**

Dissolve 0.35g of FeCl3.6H2O in 8 ml of hot water. Dissolve 0.75g of sodium  
acetate in 5ml of hot water then add 3ml of EtOH and 0.5 ml of acacH. Mix the two solutions. Cool in ice bath, filter the precipitate and dry it in air.

C5H7O2H+ NaCH3CO2NaC5H7O2+ CH3CO2H

FeCl3.6H2O+ 3NaC5H7O2Fe(C5H7O2)3+3NaCl+6H2O

Fe: [Ar]3d64S2

Fe3+: 3d54S0



 octahedral, high spin and neutral complex

|  |  |
| --- | --- |
| FeCl3.6H2O | Fe(C5H7O2)3 |
| M.wt | M.wt |
| 0.35g | T=theoretical weight |

Yield%= (observed weight/ theoretical weight)×100

Error%= {(theoretical weight- observed weight)/ theoretical weight}×100

**Experiment Nogh.7: Determination of nickel(II) in the complex  
[Ni(NH2CH2CH2NH2)3]S2O3:**

Dissolve 0.05g of the complex in 2.5ml of HCl (6M). Add 20ml of water. Heat then add 5ml of dimethylglyoxime solution. Introduce ammonia solution drop wise until red precipitate has been formed. Allow to stand for 20 minutes then filter the precipitate. Dry in an oven at 100-120C. Calculate the error.

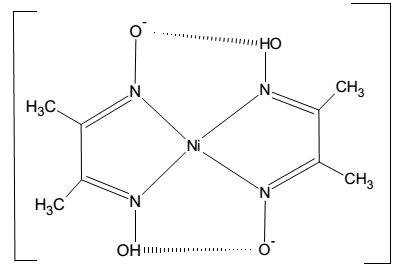
DMG= dimethylglyoxime =

;

[Ni(en)3]S2O3  Ni2+ + 2Cl- +…. .



Ni(DMG)2



|  |  |
| --- | --- |
| [Ni(en)3]S2O3 | Ni |
| M.wt | A.wt |
| 0.05g | Z |

Theortical weight percentage (T) =(Z x 100)/0.05

|  |  |
| --- | --- |
| [Ni(DMG)2] | Ni |
| M.wt | A.wt |
| red ppt. | y |

Observed weight percentage (O) =(y x 100)/0.05

E% ?

1-prepare HCl solution (250 ml, 6 M).

2- prepare DMG solution.

**Experiment No.9: Determination of oxalate in the complex  
cis-K[Cr(C2O4)2(H2O)2].2H2O:**

K[Cr(C2O4)2(H2O)2].2H2OC2O42- +K1++…..

C2O42-   2CO2+ 2e × 5

MnO41- + 8H+ +5e  Mn2+ + 4H2O ×2

2MnO41- + 16H+ +5C2O42-  10 CO2 + 2Mn2+ + 8H2O

1L1N MnO41- Ξ 1L1N C2O42-

Vml 0.1N MnO41- Ξ (V/10000) ×88/2 Ξ y g wt of C2O42- in 0.1g of the complex.

Observed weight percentage (O) =(y x 100)/0.1

|  |  |
| --- | --- |
| K[Cr(C2O4)2(H2O)2].2H2O | C2O42- |
| M.wt | 2M.wt |
| 0.1g | Z |

Theortical weight percentage (T) =(Z x 100)/0.1

Find Error%

1-Prepare 0.1N KMnO4, 250 ml.

2-Prepare 2N H2SO4 (conc. H2SO4=).

Experimental: Dissolve 0.1 g of the complex in 15ml of H2SO4(2N).Titrate with KMnO4 (0.1N) from the burette. The pink colour maintaining is indicative of reaction end point.

**Experiment No.11: Preparation of potassium bis(oxalato)cuprate(II)monohydrate:**

CuSO4.5H2O+ 4KOH+2H2C2O4K2[Cu(C2O4)2]. H2O +K2SO4+ 8H2O

Cu: [Ar]3d104S1

Cu2+: 3d94S0







|  |  |
| --- | --- |
| CuSO4.5H20 | K2[Cu(C2O4)2]. H2O |
| M.wt | M.wt |
| 0.5g | T=theoretical weight |

Yield%= (observed weight/ theoretical weight)×100

Error%= {(theoretical weight- observed weight)/ theoretical weight}×100

Experimental:

Dissolve 0.5g of CuSO4.5H2O in 20 ml of water. Dissolve KOH (0.6g) and H2C2O4 (0.5g) in 20 ml H2O. Mix the two solutions. Add 5ml of EtOH and allow to stand for 5 minutes. Filter the ppt. and dry it in air.

**Experiment No.13: Preparation of diaquabis (acetylacetonato)cobalt(II)**

**[Co(C5H7O2)2(H2O)2]:**

C5H7O2H+ NaOH NaC5H7O2+ H2O

CoCl2.6H2O+ 2NaC5H7O2[Co(C5H7O2)2(H2O)2]+2NaCl+4H2O

|  |  |
| --- | --- |
| CoCl2.6H2O | [Co(C5H7O2)2(H2O)2] |
| M.wt | M.wt |
| 0. 5g | T=theoretical weight |

Yield%= (observed weight/ theoretical weight)×100

Error%= {(theoretical weight- observed weight)/ theoretical weight}×100

Co:[Ar]3d7 4s2

Co2+: 3d7 4s0



Trans-octahedral paramagnetic complex

Experimental: Dissolve 0.2 g of NaOH in 15ml of water. Add acacH (0.5ml) and stir for 5 min. Dissolve CoCl2.6H2O (0.5 g) in water (15 ml). Mix the two solutions. Allow to stand for 30 min. Filter the precipitate and dry it in air.

**Experiment No.14: Preparation of diaquabis (acetylacetonato)nickel(II)[Ni(C5H7O2)2(H2O)2]:**

C5H7O2H+ NH4OH NH4C5H7O2+ H2O

NiCl2.6H2O+ 2NH4C5H7O2[Ni(C5H7O2)2(H2O)2]+2NH4Cl+4H2O

|  |  |
| --- | --- |
| NiCl2.6H2O | [Ni(C5H7O2)2(H2O)2] |
| M.wt | M.wt |
| 0. 5g | T=theoretical weight |

Yield%= (observed weight/ theoretical weight)×100

Error%= {(theoretical weight- observed weight)/ theoretical weight}×100

Ni:[Ar18]3d8 4s2

Ni2+: 3d8 4s0





Procedure

1. Dissolve (0.5 g) of NiCl2. 6H2O in 5 ml of water.
2. Add concentrate NH4OH dropwise until the color of the mixture becomes greenish-blue , then add 1g of acacH with stirring Cool the ppt in an ice bath then filter the ppt. Dry the ppt. in an air then calculate the yield and error.

Beer-Lam·bert law

the absorbance of light is directly proportional to the thickness of the media through which the light is beingtransmitted multiplied by the concentration of absorbing chromophore; that is, *A* = ε*bc* where *A* is the absorbance, εis the molar extinction coefficient, *b* is the thickness of the solution, and *c* is the concentration.