

Studies on Properties of Copper (II) Complex with N-Hydroxy-N- phenyl-N'-(4-Fluoro)-phenyl- (4-Chloro) Benzamidine Hydrochloride

Mrs. Sajila and Dr. (Ms.) Hemlata Mohabey*

*Department of Chemistry, Government Digvijay Post Graduate College,
Rajnandgaon (C.G.)-491-441, INDIA*

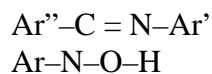
Abstract

N-Hydroxy-N-Phenyl-N'-(4- Fluoro) Phenyl-(4-Chloro) Benzamidine Hydrochloride is newly synthesized hydroxyamidine which reacts with Copper (II) at pH 2.5 to 10.5 quantitatively and forms buff coloured precipitate which is insoluble in absolute alcohol and many other organic solvent. The solid complex has molecular formula $(C_{19}H_{13}N_2OFCl)_2Cu$ which melts at $202^{\circ}C$ with decomposition. The Infra red spectra of the complex and ligand were recorded in the range $4000-450\text{ cm}^{-1}$. $C = N$ Cu bond- $N-O-Cu$ bond and formation of $Cu-N$ -is confirmed by comparison of IR spectra of the ligand and the complex. TGA studies suggest that complex is thermally stable upto $202^{\circ}C$ and melts at this temperature with decomposition. As no weight loss is observed in TG curve it is confirmed that water molecules are absent in the complex. These studies support the use of HPFPCBH for gravimetric determination of Copper in ores and alloys.

Keywords: HPFPCBH, Copper (II) IR, TGA, DTA.

1. Introduction

Hydroxyamidines are organic reagents used for detection and determination of various transition metal ions the reagent has azomethine nitrogen and $-N-O-H$ group which makes it suitable chelating agent.¹⁻⁵ The properties of the reagent can be modified by substitution in aromatic ring.



It was proposed to synthesise a new hydroxyamidine hydrochloride by condensation of N-(4-Fluoro)-phenyl-(4-Chloro) benzamidoyl chloride and N-phenyl hydroxylamine in ether medium at 0–5°C. The reagent was used to precipitate Copper (II) ion. The solid complex obtained was characterized on the basis of elemental analysis, IR. The thermal stability of the complex was evaluated by TGA studies. Volume susceptibility measurement was done by Faradays method for magnetic nature.

2. Experimental

2.1 Chemicals and Apparatus

All chemicals used were of A.R. grade. A single pan Dona balance was used for weighing purpose. pH measurements were done by systronic pH meter type 321. IR Spectra of the compound was recorded in KBr on Perkin-Elmer-1800 FTIR in the region 4000–450 cm⁻¹.

3. Method

Copper (II) solution containing 8–10 mg Copper was mixed with 1% alcoholic solution of reagent with constant stirring at pH 2.5–4.5 Heavy buff coloured precipitate was obtained which is easily filterable. It is insoluble in water and alcohol, acetone and many organic solvents. The excess of reagent was washed out with 60% alcohol water mixture. The buff coloured complex was dried at 100–120°C.

4. Elemental Analysis

The elemental analysis data support the formation of 1 : 2 complex and are in agreement with its molecular formula (C₁₉H₁₃N₂OFC1)₂Cu.

C = 59.29%, H = 3.88%, N = 7.32%, Cu = 8.42% Found

C = 61.40%, H = 3.50%, N = 7.54%, Cu = 8.56% Calculated

5. IR Spectra

The IR Spectra of the Complex was recorded in KBr on Perkin-Elmer-1800 FTIR in the region 4000–450 cm⁻¹. The weak band of ligand molecule at 2500 cm⁻¹ confirms the presence of Azomethine nitrogen.⁶ This band is absent in the IR Spectra of Copper complex confirming the involvement of this group in the formation of complex. The Free ligand has a strong band at 1640 cm⁻¹ due to C = N⁺H it is shifted to 1580 cm⁻¹ in the IR Spectra of Copper complex this confirms the presence of C = N.....Cu bond in the complex. The N–O stretching band of free ligand appears at 930 cm⁻¹ which appears at 960 cm⁻¹. The shift to higher frequency confirms the formation of –N –O–

Cu bond. According to Nakamoto⁷ metal nitrogen band appears at 400–500 cm⁻¹. This Cu–N stretching band in the present complex appeared at 460 cm⁻¹.

Magnetic Property

Only volume susceptibility⁸ measurement were done. The positive value confirms paramagnetic nature of the complex.

TGA studies

Thermo gravimetric⁹ analysis of Copper complex suggested that there is no weight loss upto 200⁰C. This confirms absence of water molecules in the Copper complex. At 210⁰C 5% weight loss is observed. Then accelerated weight loss is observed upto 590⁰C. At this temperature Cupric oxide is formed. The melting point of the complex is 202⁰C. It melts at this temperature with decomposition.

6. Conclusion

N-Hydroxy-N-Phenyl-N'-(4-Fluoro)-Phenyl-(4-Chloro) Benzamidine Hydrochloride forms buff coloured water insoluble complex. The molecular formula of the complex is (C₁₉H₁₃N₂OFCl)₂Cu m.p. of the complex is 202⁰C. The complex is paramagnetic, thermally stable, it can be easily dried. Therefore it can be used as gravimetric reagent for estimation of Copper (II) in alloys and ores.

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