

IR Spectra, Magnetic and Thermal Studies of Copper (II) Complex of N-Hydroxy-N-(4-Chloro) Phenyl N'(4-Fluoro) Phenyl Benzamidine Hydrochloride

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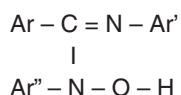
ABSTRACT

Copper (II) reacts with HCPFPBH and forms buff coloured precipitate insoluble in many organic solvents like absolute alcohol, ether, benzene, chloroform etc. The Solid complex has molecular formula $(C_{19}H_{13}N_2OFCl)_2Cu$ which melts at 202°C with decomposition. The infrared spectra of the complex was recorded in the range 4000-450 cm^{-1} . The ligand molecule consists of a weak band at 2550 cm^{-1} . This confirms the presence of azomethine nitrogen in the ligand. This band is absent in the IR Spectrum of the complex confirming the involvement of this group in complexation. The strong band of the ligand at 1640 cm^{-1} is due to C = NH⁺ group which shifts to lower frequency by 60 cm^{-1} . This confirms the formation of C = N-Cu bond. The N – O stretching mode shifts to higher frequency from 930 cm^{-1} to 960 cm^{-1} this confirms the formation of N – O – Cu bond by replacement of N – O – H proton by Copper. Cu – N band appears at 460 cm^{-1} which supports the formation of Cu – N bond. The positive value of volume susceptibility confirms that the complex is paramagnetic. TGA studies suggest that complex is thermally stable upto 202°C and melts with decomposition. At this temperature water molecules are absent which is also suggested by IR spectra of the complex. These studies support the use of N-Hydroxy-N-(4-Chloro) phenylN'(4-Fluoro) phenyl benzamidine hydrochloride for gravimetric determination of Copper (II) in ores and alloys.

Key words: Hydroxyamidine Copper Complex IR Spectra TGA

INTRODUCTION

Hydroxyamidines are organic reagents used for detection and determination of various transition metal ions.¹⁻⁵The reagent has azomethine nitrogen and –N-O-H group which makes it suitable chelating agent



The properties of the reagent can be modified by suitable substitution in aromatic ring. Therefore a new hydroxyamidine has been synthesized by condensation of N-(4-Fluoro)

phenyl benzamidoyl chloride in ether solution, like other hydroxyamidines this reagent also gives buff precipitate with Copper (II) in the pH range 2.5 to 10.5 which is easily filterable. The IR spectra, magnetic and thermal studies have been carried out.

EXPERIMENTAL

Appratus

A single pan (dona) balance was used for weighing purpose, pH of the solution was measured with systronic pH meter type 321. IR Spectra was recorded in KBr on Perkin – Elmer – 1800 (FTIR) in the region 4000-450 cm^{-1} . All the chemical used were of A.R. grade.

METHOD

Copper complex is precipitated instantaneously when reagent solution is added to cupric ions in the pH range 2.5 to 4.5. The complex is heavy, buff coloured and readily filterable. It is insoluble in water and alcohol, acetone, carbon tetra chloride and many other organic solvents. The excess of reagent is washed out easily. The complex was dried at 90°- 100°C.

The elemental analysis data support 1:2 complex

Element	Calculated %	Found %
C	61.40	59.29
H	3.50	3.88
N	7.54	7.32
Cu	8.56	8.42

The IR Spectra was recorded in KBr on Perkin-Elmer – 1800 FTIR in the region 4000-450 cm^{-1} .

The ligand molecule consists of a weak band at 2550 cm^{-1} . This confirms the presence of azomethine nitrogen in the reagent.⁶This band is absent in the IR Spectrum of Copper complex. Thus this group is involved in the complex formation.

A strong band at 1640 cm^{-1} due to $\text{C} = \text{N}^+\text{H}$ in the free ligand is shifted to 1580 cm^{-1} in the IR spectra of the complex.

This shifting of the band to lower frequency region is due to decrease in electron density in the azomethine linkage. This confirms the formation of $-\text{C} = \text{N} - \text{Cu}$ bond.

In the Copper complex N – O stretching

band appears at 960 cm^{-1} which was 930 cm^{-1} in free ligand. The shift to higher frequency confirms formation of $-\text{N} - \text{O} - \text{Cu}$ bond.

Metal - Nitrogen stretching band appears at 460 cm^{-1} . According to Nakamota this Metal - Nitrogen band appears around 400 – 500 cm^{-1} . Volume susceptibility⁸ measurements were done the positive value confirms paramagnetic nature of the complex.

TGA studies reveal that the complex starts decomposition around 202°C. There is no weight loss upto 202°C indicating that water molecules are absent in the Copper complex. At 210°C about 5% weight loss is observed. Accelerated weight loss is observed upto 590°C where CuO is formed.

CONCLUSION

IR Spectra studies confirms Cu – N bonding and Cu – O bonding. The complex formed by N-Hydroxy – N – (4-chloro) phenyl N'(4-Fluoro) phenyl benzamidine hydrochloride and Cu (II) ion. The buff coloured water insoluble complex is stable upto 202°C and melts with decomposition at this temperature. The complex is paramagnetic. Thermal stability is confirmed upto 200°C by Thermogravimetric analysis. A quick, novel method can be developed using N – Hydroxy – N – (4-Chloro) phenyl N'-(4-Fluoro) phenyl hydroxyamidine hydrochloride as gravimetric reagent for estimation of Copper (II).

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