

Antibacterial and X-ray Diffraction Study of Cr(III) and Fe(III) metal Complexes of Thiosemicarbazone Ligand

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Abstract

Comparative study of X-ray diffraction patterns of transition metal complexes like Cr (III) and Fe (III) of Thiosemicarbazone is carried out. These metal complexes has different crystal system and their x-ray diffraction studies also used for determination of various parameters such as unit cell volume and miller indices values like h, k and l. Antibacterial screening also done of these metal complexes and ligand in comparison with standard drug ciprofloxacin.

Keywords: N-4-Ethyl, propyl thiosemicarbazone, X-ray diffractogram, Biological Screening.

Introduction

Thiosemicarbazones constitute important class of drugs with several types of pharmacological actions.[1] A series of metal complexes of Cr(III) and Fe(III) with (E)-1-(2-Hydroxybenzylidene)-4-ethyl,propyl thiosemicarbazone (LH) have been synthesized and successfully characterized using various spectroanalytical techniques. The molecular structure of metal complexes was determined by X-ray diffraction studies. Additionally, ligand (LH) and all the complexes screened against antibacterial strains. Complexes were most effective against bacteria (*Staphylococcus aureus* and *Bacillus subtilis*) which are compared with standard (Ciprofloxacin). The complex forming nature of transition metal complexes with thiosemicarbazones shows antitumor activity [2,3] Thiosemicarbazone metal complexes exhibit characteristic important properties and potential biological activities. [4-8]

The X-ray diffractogram of complexes were recorded in the range of 5-80° 2θ value. The wavelengths are important part of crystal system of complexes to determine the peak position, miller (h,k,l) value, unit cell parameters and 2θ value with d radiation source of CuKα by used as X-ray diffractometer range.[9,10]

Materials and Methods

All the chemicals used were as AR grade obtained from commercial source.

Synthesis of Schiff's Base Ligand: The Schiff's base ligand (E)-1-(2-Hydroxybenzylidene)-4-ethyl,propyl thiosemicarbazone prepared by modifying reported method of scovil.[11]

Synthesis of Metal Complexes: Hot ethanolic solution of (0.01mmol) of metal salt is mixed with (0.02mmol) of solution of (E)-1-(2-Hydroxybenzylidene)-4-ethyl, propylthiosemicarbazone (LH). The reaction mixture is refluxed for 4-5 hours and allows keeping for 3-4 hours at R.T and decomposes above 300°C.

Results and Discussion

NMR Spectral Data:

The experimental assignments of the IR spectral bands to confirm the structural identity of the ligands and its metal complexes. The H-NMR spectra of Thiosemicarbazone showed one proton signal at 8.4-8.9 ppm which corresponds to azomethine linkage formed by reaction in between Salicylaldehyde and N-4 disubstituted thiosemicarbazide. It confirms the formation of Thiosemicarbazone. The H-NMR assignments are in good agreement with the values already reported.[12,13,14]

The observed NMR signals are phenolic -OH (11.79) (s), -C=N- (8.96) (s), -NH (11.06) (s), Aromatic protons 6.8-7.3 (m), -CH₂- (3.7) and -CH₃ 1.09 (t), -CH₂-(1.01) (m).

IR Spectral Data:

The IR spectral bands confirm the structural identity of Ligand and its metal complexes. According to coordination to ligand to metal ion IR spectra of ligand shows ligand should be in thione form. A sharp ν (N-H) band observed at 2976 cm⁻¹ for ONS donor ligands. A sharp ν (C=S) and low intensity δ (C=S) band observed in ligand at 1276 cm⁻¹ while in metal complexes it is in the range of 1240-1270 cm⁻¹ and 700-753 cm⁻¹. Ligand shows ν (-OH) band at 3220 cm⁻¹ due to intramolecular hydrogen bonding. Bands in the range of 420-440 cm⁻¹ due to M-O, M-N and M-S bonding.

Table 1: FTIR Spectral data of ligand and its metal complexes in (cm⁻¹)

Ligand/complex	ν (-OH)	ν (N-H)	ν (C=N)	ν (C=S)	ν (C-O)	M-S	M-N	M-O
LH	3220	2976	1538	1276,753	1186			
Cr(III)	-	-	1575	1278,701	1203	458	582	489
Fe(III)			1597	1291,707	1206	414	521	485

X-Ray Diffraction study:

X-ray diffraction study of metal complexes gives probability related to the structure of complexes. The diffractogram of metal complexes were recorded in the range of 0-80° 2 θ value and wavelength of 1.5405Å. Major reflexes were calculated and related values were determined by using Bragg's equation. All major reflections were indexed for h,k,l values using reported method.[15] Cr (III) complex having triclinic crystal system while Cr(III) complex have monoclinic crystal system. Unit cell volume for each system is determined by respected equation.

Table 2: Lattice constant, unit cell volume, crystal system and interplanar spacing of metal complexes.

Unit complex	Lattice Constant			Unit Cell Volume (Å ³)	Inter axial angle	Crystal system	2 θ	d value (Å)
	a (Å)	b(Å)	c(Å)					
Cr(III)	8.236	10.98	15.36	110.61	$\alpha \neq \beta \neq \gamma \neq 90$	Triclinic	11.90	7.43
Fe(III)	11.89	13.02	7.02	106.03	$\alpha = \beta = 90 \neq \gamma$	Monoclinic	9.18	9.66

Table 3: Miller Indices and Interplanar distance of Cr(III) complex

2 θ (obs)	2 θ (cal)	d(obs)	d(cal)	h	k	l	Intensity
9.85	9.89	8.97	8.93	0	-1	1	66.61
11.90	11.91	7.43	7.41	0	-1	2	100
12.41	12.44	7.12	7.10	1	-1	0	24.78
12.77	12.44	6.92	7.10	1	-1	0	30.60
14.51	14.20	6.08	6.22	0	0	2	22.62

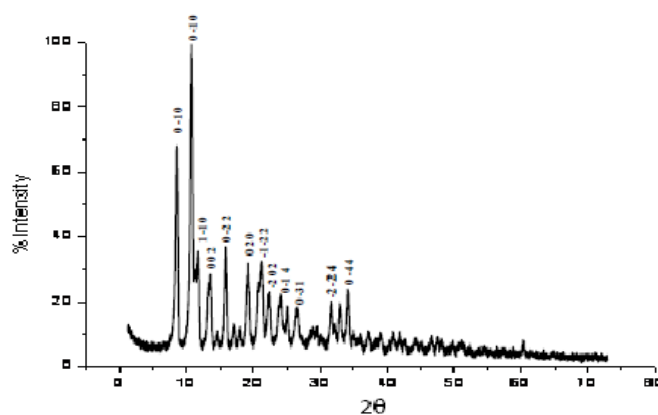


Fig.1. X-ray diffractogram of Cr(III) complex

Table 4: Miller Indices and Interplanar distance of Fe(III) complex

2θ (obs)	2θ(Cal)	d(obs)	d(cal)	h	K	l	Intensity
10.302	10.17	8.57	8.68	-1	1	0	22.54
9.18	10.17	9.66	8.68	-1	1	0	100
12.64	12.85	6.99	6.88	0	0	1	12.61
14.49	14.54	6.10	6.08	0	1	1	16
20.76	20.44	4.37	4.34	-2	2	0	11.52

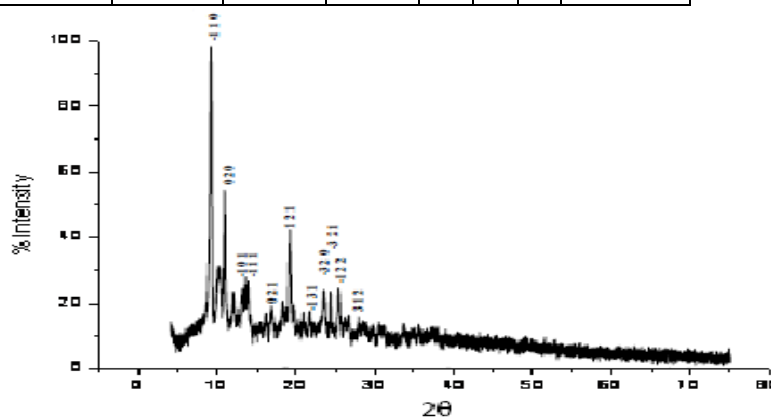


Fig.2. X-ray diffractogram of Fe(III) complex

Biological screening:

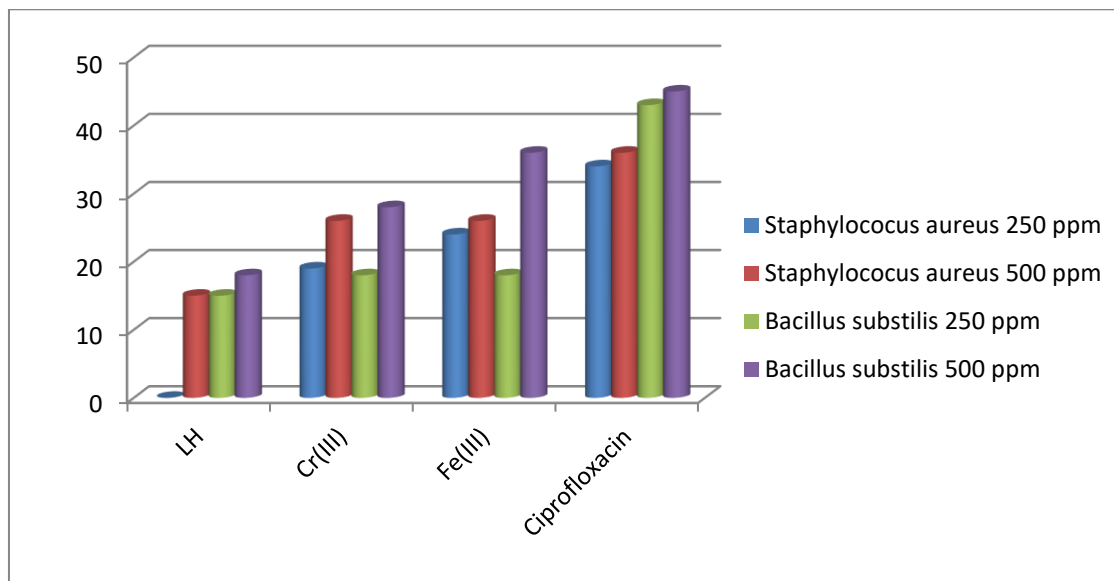
Thiosemicarbazone ligand and its Cr(III) and Fe(III) metal complexes has wide range of biological activity. [16,17] Thiosemicarbazone complexes has large pharmaceutical applications. [18] The antibacterial activity of ligand and its metal complexes are given in below table. The ligand as well as metal complexes are more active against bacteria which are compared with standard ciprofloxacin.

Table 5: Antibacterial activity of ligand and its metal complexes

Ligand/complex	Staphylococcus aureus		Bacillus subtilis	
	250ppm	500ppm	250ppm	500ppm
Ligand (LH)	00	15	15	18

Cr(III)	19	26	18	36
Fe(III)	24	26	18	36
Ciprofloxacin	34	36	43	45

Fig.3.Comparative study of antibacterial activity of Ligand and its metal complexes.



Conclusion

The Cr(III) and Fe(III) metal complexes of Thiosemicarbazone ligand were studied by x-ray diffraction method shows monoclinic and triclinic crystal system are crystalline in nature and have octahedral geometry also shows potential antibacterial activity.

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