



MULTIDISCIPLINARY RESEARCH

Prof. Rajani Shikhare

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Synthesis, IR Spectral and X-ray Diffraction Analysis of Mn (II) and Ni (II) Metal Complexes of Thiosemicarbazone Ligand.

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Abstract :

Metal complexes were prepared by the reaction of Thiosemicarbazone with $MnCl_2$ and $NiCl_2$. The Thiosemicarbazone coordinate with metal ion through the thionic sulfur, azomethine nitrogen and phenolic oxygen. The Thiosemicarbazone metal complexes were elucidated with IR and X-ray diffraction patterns. The thiosemicarbazones behaves as tridentate ligand. From analytical and spectral data the stoichiometry of the complexes has been found to be 1:2 (Metal-Ligand). The X-ray diffraction studies suggest Triclinic and Monoclinic crystal system of complexes.

Keywords: N-4-Ethyl, propyl thiosemicarbazone, X-ray diffraction.

Introduction :

Thiosemicarbazones (TSC's) are very versatile ligands. They can coordinate to metals as neutral molecules or after deprotonation as anionic ligands and can adopt a variety of different coordination modes.⁽¹⁾ Common metal complexes of thiosemicarbazones have bidentate or tridentate functions through sulfur, azomethine nitrogen and oxygen of arylidene moiety.⁽²⁾ Thiosemicarbazones obtained by condensation of N-4ethyl, propyl thiosemi-carbazide with salicylaldehyde form versatile NS/NSO chelating ligands exhibit diverse biological activities. These ligands behaves as monoanionic tridentate ligand coordinating to the metal centre through the deprotonated phenolic oxygen, thione sulfur

and azomethine nitrogen.⁽³⁾ They also coordinate as bidentate NS donor⁽⁴⁾ in spite of having phenolic oxygen as a potential donor site. This class of complex is important because of their antibacterial and antifungal,⁽⁵⁾ antimalarial, antiviral, antitumor activities.⁽⁶⁾

Materials and Methods :

All the chemicals were purchased from Sigma-Aldrich and were used as received. Melting point of ligand and metal complexes were taken in open capillary. Synthesis of (E)-1-(2-Hydroxybenzylidene)-4-Ethyl, propyl Thiosemicarbazone prepared by modifying reported method of Scovil.⁽⁷⁾

Synthesis of Metal Complexes:

Hot methanolic solution of (0.01m mol) of metal salt is mixed with (0.02mmol) solution of (E)-1-(2-hydroxy benzylidene)-4-ethyl, propyl Thiosemicarbazone (LA). A reaction mixture is refluxed for 2-3 hours and keeps at R.T for 4-5 hours. All the complexes decompose above 300°C.

Results and Discussion:

The experimental assignments of IR spectral bands are gives idea about structural identity of ligand and its metal complexes are given below in Table 1. The IR spectrum of ligand showed strong absorption bands in the range of 1500-1600 cm^{-1} which was assigned to the azomethine group, $\nu(\text{C}=\text{N})$. (8) The strong band is observed at 1290 cm^{-1} in the spectrum is due to $\nu(\text{C}=\text{S})$ and $\delta(\text{C}=\text{S})$ (9). The bands observed at 370 cm^{-1} is due to $\nu(-\text{OH})$ and $\nu(\text{N}-\text{H})$ vibrations.

It is observed that the ligand should be in the thione form. The diagnostic bands are observed in Table.1 in the spectra of metal complexes the bands due to azomethine moiety ($\text{C}=\text{N}$) is shifted to lower frequency indicating its involvement in coordination with metal ion. The $\nu(\text{C}=\text{S})$ stretching frequency also get lowered in the spectra of metal complexes shows involvement of thioke to sulfur in the coordination. The further findings such as bands in the range of 300 cm^{-1} to 470 cm^{-1} is due to M-N, M-O and M-S bonding in metal complexes. (10-15)

Table 1: FTIR Spectral data of ligand and its metal complexes in (cm^{-1})

Ligand/complex	$\nu(-\text{OH})$	$\nu(\text{N}-\text{H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{S})$	$\nu(\text{C}-\text{O})$	M-S	M-N	M-O
LA	3224	2972	1540	1290,753	1186	-	-	-
Mn(II)	-	-	1589	1278,701	1203	460	578	475
Ni(II)	-	-	1590	1270,707	1206	440	535	470

X-Ray diffraction study :

The molecular structure of metal complexes was determined by x-ray diffraction. Powder x-ray diffraction was done for metal complexes and spectra are given in Figure.1 X-ray diffraction analysis confirms which crystal system is present in metal complexes. X-ray diffraction study gives idea about structure or crystal system of metal complexes. The diffractogram of metal complexes it is in the range of 0-80°. Major reflexes were calculated and related d values were determined by using Bragg's equation. All major reflexes were indexed for h, k, l values according to the reported method.⁽¹⁶⁾

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

Unit of complex	Lattice Constant			Unit Cell Volume (Å ³)	Inter axial angle	Crystal system	2θ	d value (Å)
	a (Å)	b(Å)	c(Å)					
Mn(II)	8.146	11.44	14.40	113	$\alpha \neq \beta \neq \gamma \neq 90$	Triclinic	7.28	12.18
Ni(II)	11.90	14.00	7.12	106.9	$\alpha = \beta = 90 \neq \gamma$	Monoclinic	9.30	10.00

Table 3: Miller Indices and Interplanar distance of Mn (II) complex

2θ (obs)	2θ(cal)	d(obs)	d(cal)	h	k	l	Intensity
7.28	7.18	12.18	12.29	0	0	1	100
18.62	18.08	4.76	4.82	-1	-1	2	35.98
20.62	20.64	4.28	4.29	-1	-2	1	42.11
16.94	16.90	5.22	5.24	0	-2	2	46.23
16.61	16.86	5.33	5.25	1	0	2	38.40
10.87	10.84	8.12	8.15	-1	0	0	74.94

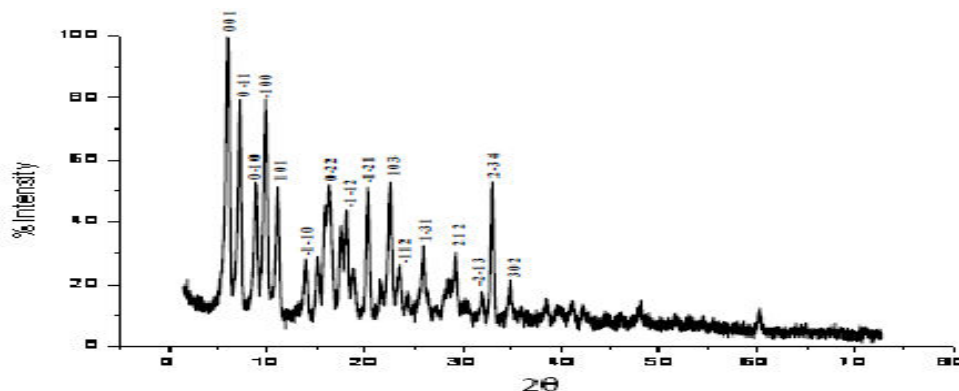


Fig.2. X-ray diffractogram of Mn(II) complex.

Table 4: Miller Indices and Interplanar distance of Ni(II) complex.

2θ (obs)	2θ (Cal)	d (obs)	d (cal)	h	k	l	Intensity
9.30	10.11	9.80	8.50	-1	1	0	100
21.35	21.12	4.15	4.18	1	2	1	36.71
14.05	13.58	6.29	6.51	0	2	0	14.67
14.87	15.17	5.96	5.83	-1	1	1	16.41
18.54	18.74	4.77	4.71	0	2	1	9.94
20.76	20.44	4.27	4.34	0	3	0	8.44

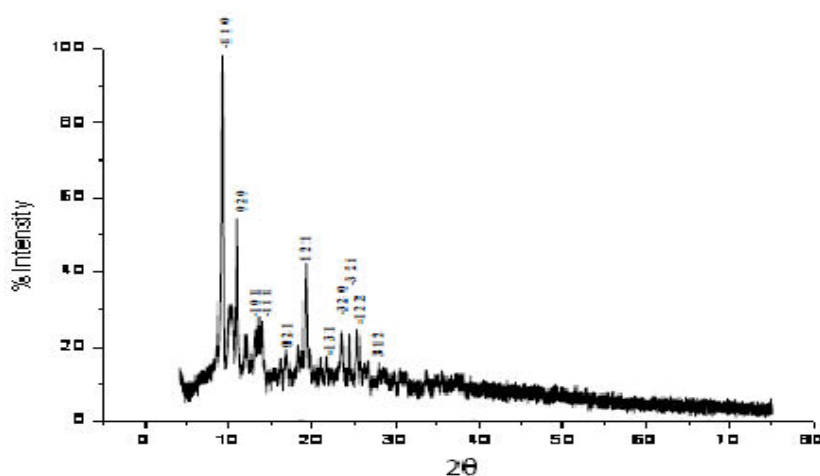


Fig.2. X-ray diffractogram of Ni (II) complex.

Conclusion:

The Mn(II) and Ni(II) metal complexes of Thiosemicarbazone ligand were studied by X-ray diffraction shows Triclinic and Monoclinic crystal system are crystalline in nature and have octahedral geometry and their structures were confirmed by IR spectral assignments.

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