CODEN [USA]: IAJPBB ISSN : 2349-7750

INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES
SJIF Impact Factor: 7.187

Available online at: http://www.iajps.com Research Article

X-RAY DIFFRACTION STUDIES OF NEW HYDRAZONE SCHIFF BASE LIGAND AND DIVALENT Mn (II), Ni (II) AND Zn (II) COMPLEXES

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Article Received: January 2021 **Accepted:** February 2021 **Published:** February 2021

Abstract:
The divalent trivalent metal complexes of were synthesized by the reaction of new heterocyclic hydrazone Schiff base ligand of 4-(diethylamino)-2-hydroxybenzylidene)-4-oxopiperidine-1-carbohydrazide (H_2L): The divalent metal (II) complex were spectrochemically analyzed viz., elemental analysis, Fourier Transform Infrared, Ultraviolet- visible spectroscopy, and X-ray diffraction. The ratio of stoichiometry of ligand with Mn(II), Ni(II) and Zn(II) ion were observed as 1:1 (M:L) in their complexes. On the basis of spectral data the complexes were suggested a octahedral structure to the Mn(II),Ni(II),Zn(II) ions.

KEYWORDS: *Heterocyclic hydrazone, metal(II) complexes, spectrochemical analysis, X-ray diffraction.*

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Please cite this article in press Gajanan Dongare, ***X-Ray Diffraction Studies Of New Hydrazone Schiff Base Ligand And Divalent Mn (II), Ni (II) And Zn (II) Complexes.,*** *Indo Am. J. P. Sci, 2021; 08(02).*

1. INTRODUCTION:

The hydrazone Schiff base ligands are one of the most broadly classes of ligands in coordination chemistry. The hydrazone Schiff bas ligands and their derivatives are the useful synthons for the various heterocyclic with heteroatoms in five, six or seven-membered aromatic rings containing compounds [1-5]. The hydrazone ligands are imparts their own functionality and can tune a unique properties of the overall in the complexation due to the structure and bonding. The hydrazone and their derivatives are highly important because of these ligands developed due to their diverse chelating ability, structural flexibility and pharmacological activities such as antimicrobial, anti-inflammatory, analgesic, antifungal, antiviral, anticancer and anti-tuberculosis. In addition to this, some transition metal complexes are important in catalysis, materials synthesis, photochemistry and biological systems [6-8]. The medicinal inorganic chemistry can exploit the unique properties of metal ions for the designing of new hydrazone drugs. The hydrazone and their complexes were used in the medicinal purposes due to the metal-ligand interactions influence ligand exchange reactions. Hydrazone compounds was synthesized by coupling methods and now using in medical biotechnology to couple drugs to targeted antibodies e.g. antibodies against the certain type of cancer cell. The phenomenon in which hydrazone bond is stable at neutral pH in the blood stream, but is bond destroyed in the acidic condition of lysosomes of the cell. The drug is thereby released in the cell where it is exerts in its function [9-12].

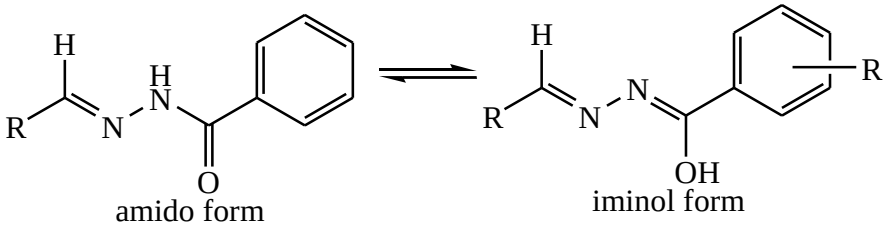
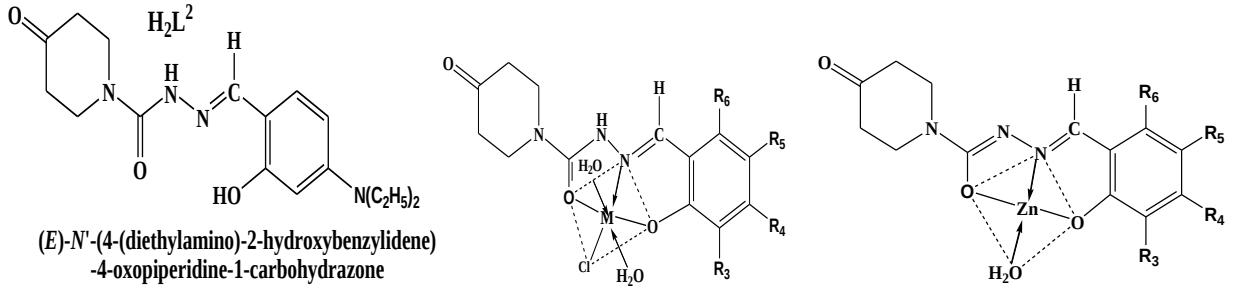


Fig.1 Tautomerism in hydrazone Schiff ligand

2. EXPERIMENTAL:

2.1 SYNTHESIS OF METAL COMPLEXES:

The synthesis of ligand and its metal (II) complexes was reported in *Gajanan Dongare etl.* [13-14]. A warm solution of Schiff base of 4-(diethylamino)-2-hydroxybenzylidene)-4-oxopiperidine-1-carbohydrazide (H_2L) in equimolar amounts was reacted to respective metal chlorides of $MnCl_2.2H_2O$, Ni(II)$Cl_2.6H_2O$ $ZnCl_2.4H_2O$ to obtained metal complexes.



(a) (b) (c)

Where R_3,R_5,R_6=H, and R4=N(C_2H_5)$_2$

Fig.2. Structure of (a) hydrazone ligand H_2L (b) Metal complex of Mn (II), Ni(II) complex (c) Zn(II) complex

3. RESULTS AND DISCUSSION:

3.1 X-ray diffraction study of metal complexes:

The X-ray diffractograms of ligand [H_2L] and its Mn(II), Ni(II) and Zn(II) complexes are recorded to determine the types of crystal system, lattice parameters and cell volume Fig.3 (a-d) and data listed in table 1-4.The values of unit cell parameters for ligand and its Mn(II), Ni(II) and Zn(II) complexes are as follows; ligand [H_2L]: Bravais crystal system are representing the monoclinic system, a = 12.013 Å, b =6.5415 Å, c =13.35353 Å and α = γ = (90 deg.), β= (95 deg.), (V) = 1052.223 Å^3 and interplanar spacing from peak position = 3.66430Å, Space group=P with Z=2 [15-

20].[Mn(HL)Cl(H_2O)$_2$] complex: crystal system is monoclinic system, a = 12.013 Å, b = 6.5415 Å, c = 13.3553 Å, and α = γ = 90 deg., β= (95 deg.), (V)= 1043.687Å^3, Space group=P with Z=2;[Ni(HL)Cl(H_2O)$_2$]: Crystal system is orthorhombic, a = 8.0190 Å, b = 8.0130 Å, c = 3.6610 Å and α = β = γ (90 deg.), (V)= 235.242Å^3, Space group=C with Z=2 [169-170]. [Zn(L)(H_2O)$_2$] complex: crystal system is orthorhombic, a = 7.7800 Å, b = 8.5000 Å, c = 8.0800 Å and α = β = γ = (90 degree),V = 534.330 Å^3, Space group=I with Z=4.The average crystallite particle size of ligand (H_2L), Mn(II), Ni(II) and Zn(II) complexes was 21.53, 37.030, 39.4341 and 34.89 nm, respectively, which indicating the nano crystalline nature [21-24].

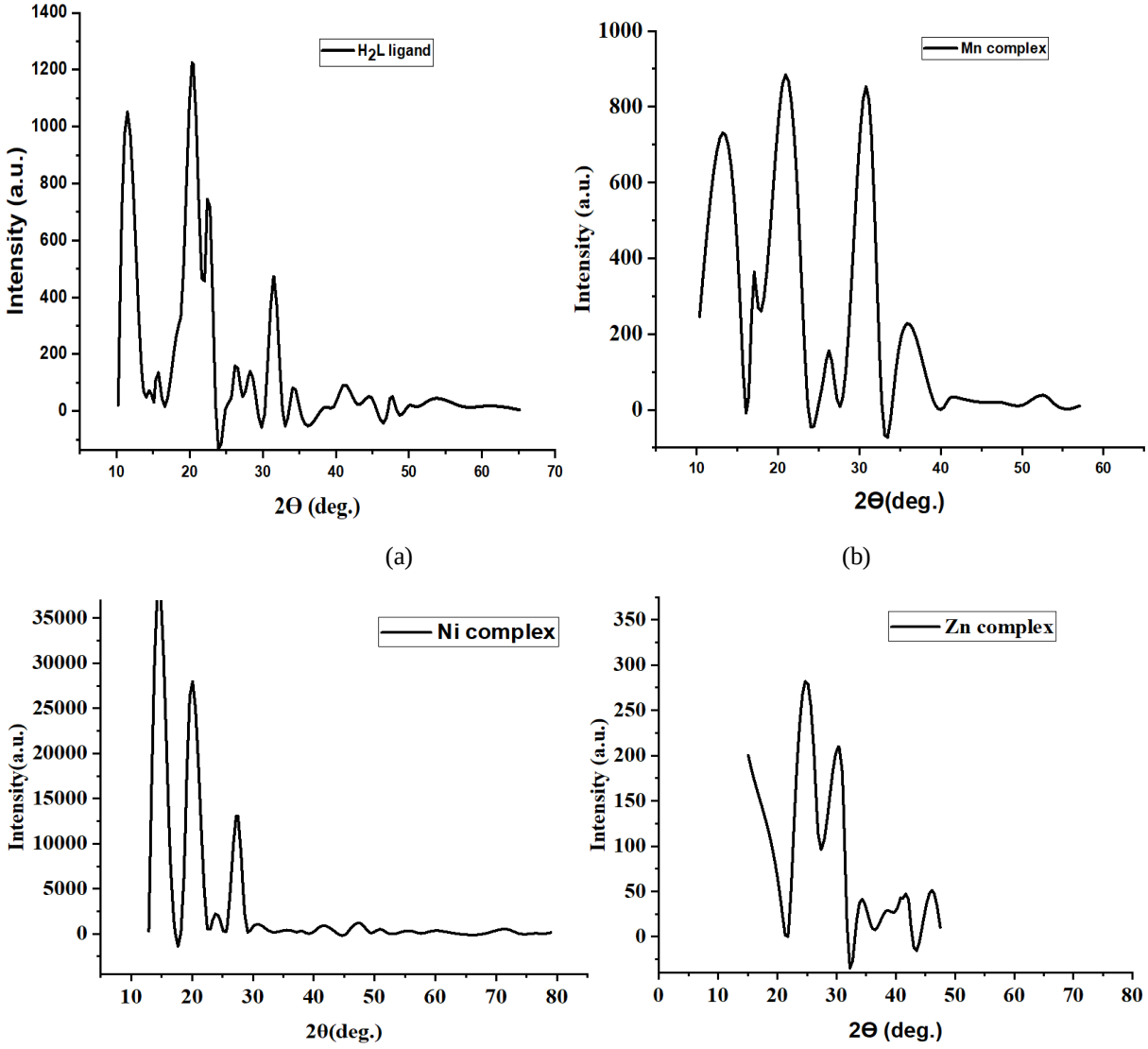


Fig.3 XRD spectra of (a) Ligand, Mn(II), Ni(II) and Zn(II) complexes

Table 1. Indexed X-ray diffraction data of (DEHOPC)-H_2L ligand

Peak No.	2Θ (Obs)	d (Obs)	2Θ (Cal)	d (Cal)	Intensity	Relative Intensity (%)	FWHM (Deg.) β	Miller Indices h	k	l
1.	10.328	8.558	10.42	8.482	179	14.67	0.52	1	0	1
2.	13.7	6.457	13.29	6.654	68.8	5.64	0.75	0	0	2
3.	14.801	5.98	14.76	5.995	58	4.75	0.46	2	0	0
5.	15.364	5.763	15.39	5.754	110	9.02	0.27	1	1	-1
6.	16.24	5.453	16.46	5.38	42	3.44	0.28	2	0	2
7.	18.53	4.783	18.853	4.7	304	24.92	0.27	1	1	-2
8.	18.799	4.717	18.98	4.671	329	26.97	0.23	0	2	0
9.	20.502	4.3285	20.6	4.309	1220	100.00	0.299	1	0	3
10.	22.153	4.0094	22.06	4.027	519	42.54	0.309	2	1	-2
11.	22.406	3.9648	22.54	3.942	744	60.98	0.177	3	0	-1
12	25.79	3.451	25.94			5.16	0.41	3	1	0
13	26.213	3.397	26.18	3.415	158	12.95	0.621	1	1	3
14	28.28	3.1532	28.27	3.115	139	11.39	0.38	3	1	1
15	31.544	2.8339	31.53	2.535	472	38.69	0.362	2	2	-1
16	34.099	2.6272	34.13	2.625	81	6.64	0.255	1	0	5
17	41.038	2.1976	41.16	2.189	91	7.46	0.66	1	1	-1
18	52.827	1.7316	52.11	1.72	38.9	3.19	0.161	1	1	4

Unit cell data and its crystal lattice parameter

a = 12.013Å	V	: 1052.223Å^3
b =6.5415Å	λ	: 1.54059 nm
c =13.3553Å	Crystal System	: Monoclinic
α =90 deg.	Nature	: Crystalline
β =95 deg.	Average Particle Size	: 21.53 nm
		Space Group=P
γ =90 deg.	Z=2	

Interplanar spacing from peak position = 3.66430Å

Table 2. Indexed X-ray diffraction data of [Mn (HL)(H_2O)$_2$(Cl)] complex

Peak No.	2Ɵ (Obs)	d (Obs)	2Ɵ (Cal)	d (Cal)	Intensities	Relative Intensities (%)	FWHM (Deg) β	Miller Indices h	k	l
1.	10.355	8.536	10.45	8.46	246	28.51	0.142	1	0	1
2.	15.349	5.768	15.08	5.869	274	31.75	0.252	1	1	-1
3.	17.073	5.1894	16.51	5.365	364	42.18	0.152	2	0	-2
4.	17.22	5.1453	17.12	5.175	334	38.70	0.139	0	1	2
5.	18.695	4.7426	18.91	4.688	382	44.26	0.238	2	1	0
6.	20.587	4.3108	20.64	4.299	863	100	0.327	1	0	3
7.	22.509	3.9469	22.62	3.927	526	60.95	0.246	3	0	-1
8.	25.561	3.4821	25.05	3.552	78	9.04	0.217	0	0	4
9.	26.259	3.3911	26.18	3.401	156	18.08	0.276	0	2	0
10.	28.339	3.1468	28.39	3.142	104	12.05	0.337	0	2	2
11.	31.49	2.8387	31.03	2.88	725	84.01	0.222	4	1	-2
12.	34.455	2.6009	34.45	2.601	99	11.47	0.177	3	2	-1
13.	45.858	1.9772	45.86	1.977	19.3	2.24	0.19	2	0	6
14.	47.374	1.9174	47.39	1.917	19.6	2.27	0.205	4	2	3

Unit cell data and its crystal lattice parameter

a = 12.013Å	V	: 1043.687Å^3
b =6.5415Å	λ	: 1.54059 nm
c =13.3553Å	Crystal System	: Monoclinic
α =90 deg.	Nature	: Crystalline
β =95 deg.	Average Particle Size	: 37.030 nm
γ =90 deg.	Z=2	Space Group=P

Interplanar spacing from peak position = 3.45080Å

Table 3. Indexed X-ray diffraction data of complex[Ni(HL)Cl(H_2O)$_2$]

Peak No.	2Θ (Obs)	d (Obs)	2Θ (Cal)	d (Cal)	Intensity	Relative Intensities	FWHM (Deg.) &	Miller Indices h	k	l
1.	15.685	5.6454	15.65	5.668	346	100.00	0.177	1	1	0
2.	22.225	3.9967	22.15	4.01	195	56.36	0.216	0	2	0
3.	29.03	3.073	29.01	3.075	35	10.12	0.22	0	0	1
4.	31.679	2.8221	31.54	2.834	20.8	6.01	0.138	1	1	1
5.	32.496	2.7531	33.11	2.704	25.8	7.46	0.138	2	2	0
6.	33.159	2.6995	33.12	2.704	245	70.81	0.238	0	2	1
7.	35.46	2.5293	35.37	2.536	62	17.92	0.20	3	1	0
8.	40.268	2.2378	40.21	2.241	105	30.35	0.164	2	2	1
9.	43.498	2.0788	43.37	2.084	61	17.63	0.284	1	3	1
10.	43.934	2.0592	43.39	2.084	10	2.89	0.26	4	0	4
11.	45.358	1.9978	45.32	2.003	52.30	15.12	0.293	3	3	0
12.	49.58	1.8371	49.77	1.831	30	8.67	0.31	0	0	2
13.	50.2	1.8159	50.89	1.093	1.30	0.38	0.10	2	4	0
14.	51.15	1.785	51.96	1.758	6.00	1.73	0.20	4	0	1
15.	54.762	1.6749	54.62	1.679	44.70	12.92	0.186	3	3	1
16.	57.26	1.6077	57.19	1.61	57	16.47	0.26	4	2	1
17.	58.985	1.5647	58.7	1.572	14	4.05	0.27	5	1	0

Unit cell data and its crystal lattice parameter

a =	V	: 235.242Å^3
b	λ	: 1.54059 nm
c	Crystal System	: Orthorhombic
α =90 deg.	Nature	: Crystalline
β =90 deg.	Average Particle Size : 39.4341 nm	
γ =90 deg.	Z=2	Space group=C

Interplanar spacing from peak position = 2.2066Å

Table 4. Indexed X-ray diffraction data of [Zn(L)(H_2O)] complex

Peak No.	2Θ (Obs)	d (Obs)	2Θ (Cal)	d (Cal)	Intensities	Rel.Intensities (%)	FWHM (Deg) β	h	k	l
1.	15.079	5.8705	15.12	5.856	200	95.69	0.219	0	1	1
2.	15.401	5.7486	15.43	5.739	191	91.39	0.211	1	1	0
3.	20.839	4.259	20.88	4.25	25.70	12.30	0.218	0	2	0
4.	22.8	3.8972	22.84	3.89	122.60	58.66	0.240	2	0	0
5.	26.251	3.3921	26.3	3.386	195	93.30	0.186	1	1	2
6.	26.907	3.3109	26.97	3.304	112.70	53.92	0.227	1	2	1
7.	27.452	3.246	27.5	3.24	96.70	46.27	0.231	2	1	1
8.	30.442	2.934	30.5	2.928	209	100	0.212	0	2	1
9.	31.101	2.8733	31.14	2.869	160	76.56	0.201	2	2	0
10.	38.324	2.3467	38.45	2.339	28.30	13.54	0.287	2	2	2
11.	40.447	2.2283	40.55	2.33	35.40	16.94	0.214	1	3	2
12.	40.829	2.2084	40.92	2.203	43	20.57	0.211	1	3	2
13.	41.18	2.1904	41.31	2.184	42	20.10	0.250	2	3	1
14.	42.181	2.1407	42.14	2.143	39	18.66	0.253	1	2	3
15.	44.69	2.0262	44.74	2.114	19.20	9.19	0.290	3	2	1
16.	46.49	1.9518	46.48	1.952	47.80	22.87	0.384	0	4	0
17.	48.264	1.8841	48.36	1.181	37.50	17.94	0.241	3	3	0
18.	52.24	1.7496	52.4	1.762	28.70	13.73	0.249	4	1	1
19.	53.973	1.6975	53.54	1.71	151.40	72.44	0.210	2	4	2

Unit cell data and its crystal lattice parameter

a = 7.7800Å	V	: 534.330Å^3
b =8.5000Å	λ	: 1.54059 nm
c =8.0800Å	Crystal System	: Orth-rhombic
α =90 deg.	Nature	: Crystalline
β =90 deg.	Average Particle Size : 34.89 nm	
γ =90 deg.	Z=4	Space Group=I
Interplanar spacing from peak position = 2.5623 Å		

3.4 Correlation between ionic radii and activation energy (Ea) of metal complexes of hydrazone ligand:

In the present investigation, we have been studied the correlation of ionic radii (pm) with activation energy (Ea =kJ/mole) for the accounting of the increase or decrease in the activation energy (Ea) with divalent and trivalent metal complexes. The ionic radii of transition metal ions (M^{2+} and M^{3+}) was reported by Linus Pauling (1927), R.D.Shannon and C.T.Prewitt (1969) and their literature values are (pm) Cr(III)=62, Mn(II)=80, Fe(III)=64, Co(II)=71, Ni(II)= 59,Cu(II)= 63, Zn(II)=73 and these values are considered for the correlation with the activation energy [66-70].The correlation between ionic radii with average value of activation energy (E_a) of metal complexes of [H_2L] ligands are represented in **fig.6** and their data summarized in **table 5**.

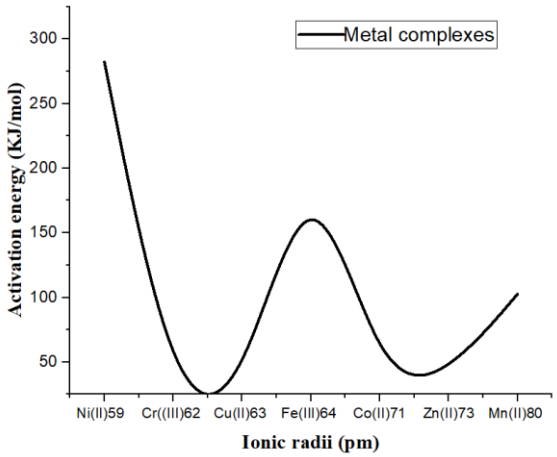


Fig.3 Correlation between ionic radii and average activation energy (Ea.) of metal complexes

Table 5. Correlation between ionic radii and average activation energy (Ea.) of metal complexes

Metal ions	Cationic radii Pauling scale and Shandon's radii (pm)	Average Activation energy (Ea.) of metal complexes (KJ mol^{-1})
Ni(II)	59	282.22
Cr(III)	62	58.30
Cu(II)	63	51.93
Fe(II)	64	160.12
Co(II)	71	64.25
Zn(II)	73	48.60
Mn(II)	80	102.56

CONCLUSION:

The hydrazone ligand of 4-(diethylamino)-2-hydroxybenzylidene)-4-oxopiperidine-1-carbohydrazide and its metal complexes of Mn (II),Ni(II),Zn(II) were analyzed by spectro-analytical techniques. The average crystallite particle size of ligand (H_2L), Mn(II), Ni(II) and Zn(II) complexes were indicating the nano crystalline nature.

ACKNOWLEDGEMENTS:

The author are thankful to authorities of SGB Amravati University for providing necessary laboratory facilities, and grateful to Principal, Shri Shivaji Science College, Amravati for recording XRD spectra.

Funding: This research work was not funded by any government or non-government authorities.

Disclosure statement: The author have no conflict of interest.

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