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SYNTHESIS OF NEW MONONUCLEAR METAL COMPLEX OF MEDICINAL SIGNIFICANCE FROM HYDRAZONE SCHIFF BASE: SPECTROSCOPIC CHARACTERIZATION, AND ANTIMICROBIAL STUDY

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Abstract:
The [Zn(L)(H_2O)]complex was synthesized by the condensation reaction of new medicinal significance hydrazone base ligand of 5-bromo-2-hydroxybenzylidene)-4-oxopiperidine-1-carbohydrazide (H_2L). The complex was characterized by elemental and spectral techniques such as Infrared, UV-vis, and molar conductance, magnetic susceptibility, powder XRD. The ligand and metal stoichiometric ratio was found 1:1 (M:L) in the complex. The spectral, TG data shows tetrahedral geometry toward Zn (II) complex. X-ray diffraction study confirms nanocrystalline nature of metal complex.

Keywords: *Mononuclear, metal complex, spectral characterization, X-ray diffraction, Antimicrobial study.*

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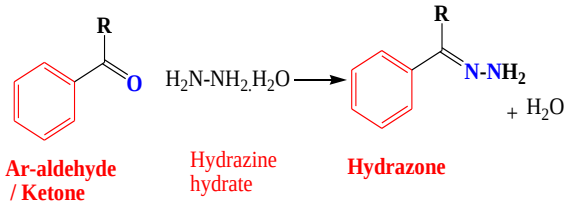
1. INTRODUCTION:

The derivatives of hydrazide-hydrazones are gain enormous importance in pharmaceutical and medicinal field due to its versatile characters playing an important role in synthesis and its application. The researchers have been attracted in large number over the years because of their promising biological activities viz., antimicrobial, anticancer, antituberculosis antiviral and anticonvulsant activities. The most of hydrazide-hydrazone derivatives have been considered as medicinal drugs and have been routinely used in clinics viz., nitrofurazone, furazolidone, nitrofurantoin. Adamantine derivatives are important constituents in medicinal fileld due to their variable bioactivities. In the year of 1960s, adamantine derivative found in amantadine treated as for antiviral activity. A number of hydrazide and hydrazone derivatives and its Zn(II) complex to find new compounds for the biological activities. As a result, the adamantane derivatives were discovered with enormous biological activities, such as antiviral, antimicrobial, anticancer angiogenesis inhibition, anti-inflammation,11β hydroxysteroid dehydrogenase type 1 (11β-HSD1) inhibition, and tyrosinase inhibition activities [1-6].



Scheme1. Synthesis of hydrazone

The azomethine (H-C=N) bond are versatile and playing important role in Schiff bases ligands for coordination chemistry with metal complexes. In recent years, metal (II) complexes of hydrazone Schiff bases have been attracted attention due to their interesting nanocrystalline, antimicrobial activity. The hydrazone Schiff base complexes derived from Salicylaldehyde and its derivatives with primary amines, bearing the ONO donor sets of atoms have particular biological activities [7–10]. The tridentate hydrazone is a special types Schiff base ligand enhancing the peculiar biological activities [11–14]. In the present research work, new $[Zn(L)(H_2O)]$ was prepared where H_2L is the dibasic tridentate form is reported herein.

2. EXPERIMENTAL:

2.1 SYNTHESIS OF Zn(II) COMPLEX:

The equimolar quantity of a hot solution of hydrazone Schiff base (H_2L) in DMSO was refluxed to the hot alcoholic solution of $ZnCl_2.6H_2O$. The p^H of the solution was maintained 7.5 with a mixture of alcohol and ammonia solution (1.5 ml). The product was recovered by the vacuum filtration and later to wash the small quantity of diethyl ether, and finally by warm ethanol. The elemental data, formula, mol.wt, and molar conductance data of metal complexes are summarised in (**Table 1**).

2.2 MATERIALS:

The chemicals used in the present research work were of anal. grade from S.D. fine chemicals, and Hi-media. The solvents were dried through the distillation. The silica granules of size 60-100 mess were used to remove the water molecule from solvents.

2.3 ANALYSIS AND PHYSICAL MEASUREMENTS:

Infrared (IR) spectra was recorded on Adv. Bruker spectrophotometer. Elemental (C, H, and N) analysis were made on a on the Carlo-Erba analyzer. The measurement of magnetic susceptibility was performed on a Sherwood magnetic susceptibility balance (MK-1). The ^1H and ^{13}C-NMR spectrum of ligand in DMSO-d_6 in (TMS) on a Bruker advance 500 MHz spectrophotometer. The (ESI) mass spectrum of hydrazone ligand was measured on micro-mass spectrophotometer. The double beam (1800) Shimadzu UV-visible spectrophotometer was used to record absorption spectra in the range of 200-1100 nm. The simultaneous TG-DTG curves were recorded on Perkin-Elmer (TGA-4000) analyzer in 40 - 800ºC with the thermal rate of 20 ^{0}C per min in N_2 atmosphere. X-ray diffraction (pattern) were measured on Rigaku-Miniflex (600). The M.P. was determined on electrical melting point apparatus. The molar conductivities of metal complexes were carried out by using Elico conductivity meter-180.

Table1.Analytical data of hydrazone Ligand (H_2L) and its Zn(II) complex

Compounds	Molecular Formula	Mol.wt.	Elemental analysis found (calcd.) %					Molar cond. (Sm^2mol^{-1})
			C	H	N	Br	M	
[H_2L] Ligand	$C_{13}H_{14}N_3O_3Br$	340.17	46.02 (45.90)	4.25 (4.14)	12.40 (12.35)	23.70 (23.48)	---	---
[Zn(L)(H_2O)]	$ZnC_{13}H_{14}N_3O_4Br$	421.55	37.20 (37.03)	3.65 (3.34)	9.98 (9.96)	18.90 (18.95)	15.60 (15.50)	22.30

3. RESULTS AND DISCUSSION:

3.1 IR spectral characterisation of Zn (II) complex:

Infrared spectroscopy consist of the measurement of the interaction of IR radiation with the compounds by the absorption of radiation. The infrared spectrum of an organic and inorganic compound provides an excellent "fingerprint" which imparts more characteristic to identify number of functional groups such as carbonyl, imino,(C=C), (C-C) bonds in the compound, crystal lattice, halide linkage, tautomerism, enolic and phenolic group of vibration. Zn(II) complexes indicate enolization occurred by the coordination to the metal ion after deprotonation. The appearance of non-ligand bands about at 824-890 cm^{-1} inferred that the presence of H_2O molecule and supported to TG analysis and the new bands in the regions 504-575 and 430-461 cm^{-1} indicating M-O and M-N bond vibrations respectively [15-20]. On the basis of the IR spectral data of (Table 2) and Figure (a) Ligand and (b) Zn(II) complex showing the phenolic-OH deprotonation (monobasic), bonding with N in azomethine and O in carbonyl with metal ions suggested the ligand possesses tridentate moiety, and is mononuclear in nature.

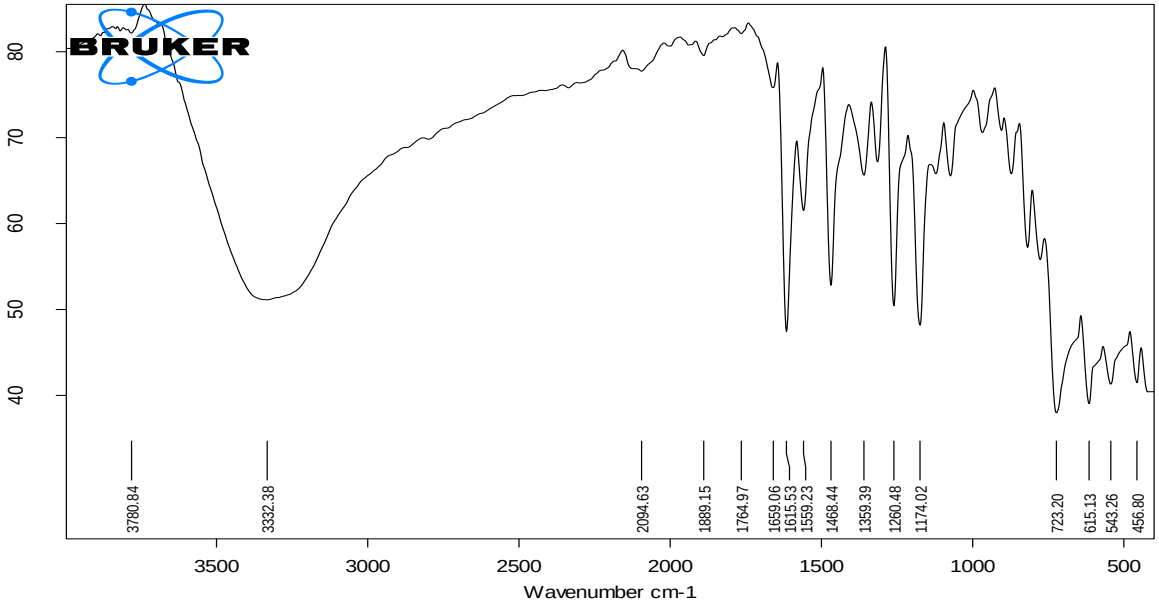


Fig.1 FT-IR spectrum of Ligand

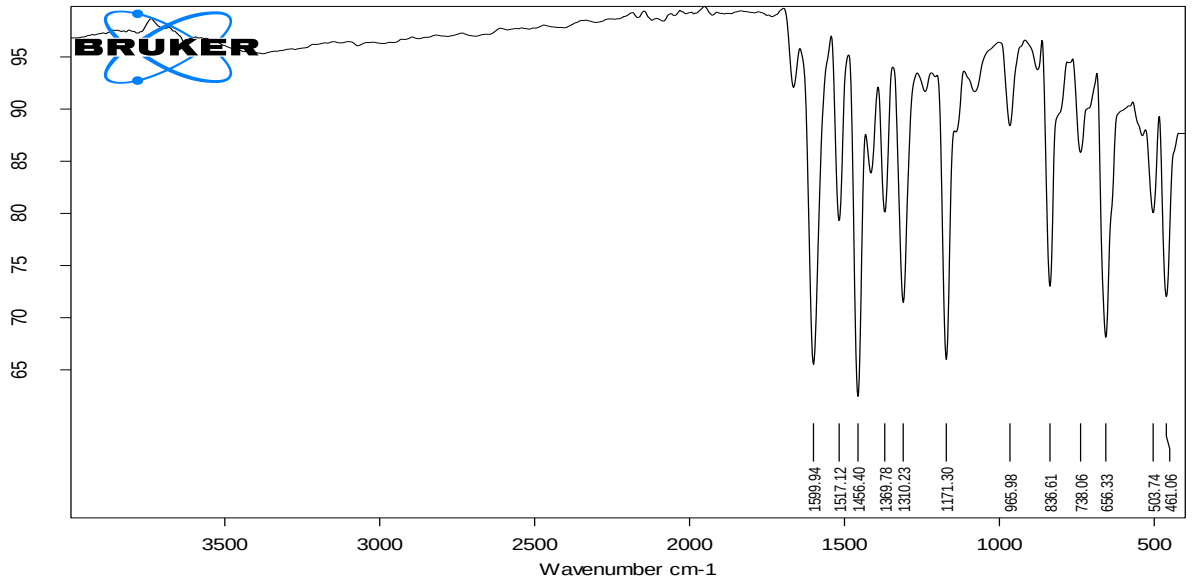


Fig.2 FT-IR spectrum of Zn(II)complex

Table 2. FT-IR spectral data of hydrazone ligand (H_2L) and Zn(II) complex (cm^{-1})

Compound (cm^{-1})	*ν*(C=N)	*ν*(C-O)	*ν*(N-N)	ν(H_2O)	*ν*(M-O)
(H_2L) ligand	1625	1230	950	---	---
[Zn(L)(H_2O)]	1576	1380	966	890	504

3.2 Magnetic moment, and UV-Visible spectroscopic studies:

The Zn (II) complex exhibits of the three important absorption of spectral band at 367,294,232 nm are due to intra ligand charge transfer (LMCT and ILCT) at degenerate level. The complex was found diamagnetic at room temperature, suggested and may have tetrahedral geometry to Zn(II) complex [21-24].The absorbance and magnetic moment data Of complex shown in Table (3).

Table 3. Solution conductivity, Magnetic moment, Electronic spectral data of Zn(II) complex

Compounds	Molar cond. (S m^2mol^{-1})	Magnetic Moment μ_{eff} (B.M.)	Absorption (λ=nm)	Spectral Assignments
[Zn(L)(H_2O)]	22.30	Diamagnetic	367 294 232	*LMCT* *ILCT (n→ π*)* *ILCT (π→ π*)*

3.3 X-ray diffraction (powder) study:

The X-ray diffractograms of of Zn(II) complex was performed to determine for evaluation of the types of crystal system, lattice parameters and cell volume and spectra in Fig.(3) and data given in Table 4. The X-ray diffraction pattern was studied using source target CuKa radiation = 1.540559 A˚ to find out compounds nature, peak intensity, crystallite size, and unit cell dimensions. The Bragg's relation (nλ = 2dsinθ) was employed for the determination of angle (2θ) and interplanar distance (d) [24-27]. It provides information of unit cell dimensions, characterization of crystalline materials, and identification of fine-grained determination and unit cell dimensions, measurement of sample purity. The crystalline substance possesses the three-dimensional diffraction for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is technique for

the study of crystal structures and atomic spacing of inorganic complex compound. Miller indices were calculated and found in expected directions which are supported to the crystal lattice parameter of h, k, l values. The Zn(II) complex crystal system is monoclinic, a = 7.7800Åb =8.5000Å, c =8.0800Å, and α = β=γ = 90deg.,V = 534.330Å^3, Space group=P with Z=1 [25-34].The full width at half maximum intensity (FWHM), the average crystallite particle size Zn(II) complex was 22.12nm which indicating to their nano crystalline nature.

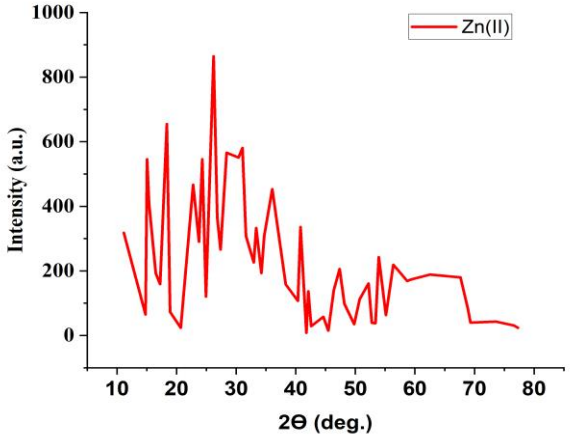


Fig.3 X-ray Diffraction pattern of Zn(II) complex

Table 4- Indexed X-ray diffraction data and lattice parameter of Zn(II) complex

Crystal parameters	Zn(II) complex
Empirical formula	$ZnC_{13}H_{14}N_3O_4Br$
Formula weight	421.55
Temperature (K)	301k
Crystal system	Monoclinic
Scan speed time	10 deg/min.
X-ray	40 kV, 15 mA
Refinement/ Scan range	10-80 deg.
Space group	P 1 21/n 1(14)
a	a = 7.7800Å
b	b =8.5000Å
c	c =8.0800Å
α (°)	α =90deg.
β (°)	β =90deg.
γ (°)	γ = 90deg.
Volume (Å^3)	534.330 Å^3
Z	1
Radiation (λ)	CuKa 1.54059 nm
2 Θ range for data collection (°)	11.28 - 63.05
I/Ic (RIR)	0.85
FWHM (β)	0.22-0.81
Average particle size	22.12nm

4. ANTIMICROBIAL STUDY:

The Microbes are very causative components for different types of diseases viz., pneumonia, ameobiasis, typhoid, malaria, and also some diseases i.e tuberculosis, influenza. The infectious diseases caused by the bacteria affect of people and are leading to causes of the death. The antimicrobial agents have been incorporated at a rate exceeding the ability to integrate them into clinical practice. The several advancements in the development of antimycotic agents, antifungal chemotherapy remains problematic in many cases and search for new antifungal agents continues to be an inevitable task. In spite of the attempts to generate new antimicrobial agents, many problems remain to be solved for currently available antimicrobial drugs. Th hydrazone-hydrazide Schiff bases the most versatile component of these compounds against microbes, and therefore, are useful as microbial candidate in biological sciences.

CONCLUSION:

The hydrazone ligand and its Zn(II) complex was synthesized by single step reaction. The mononuclear complex of the type $[Zn(L)(H_2O)]$ have been characterized by spectro-analytical techniques. Molar conductance parameters inferred that the complexes are non-electrolytic behaviors. The nano crystalline nature of Zn(II)complex was conferred by XRD diffraction techniques.

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