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Research Article

**SPECTRO-ANALYTICAL EVALUATION OF NEW
HYDRAZONE SCHIFF BASE AND ITS DIVALENT COPPER (II)
COMPLEX AND FLUORESCENCE STUDY**

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Abstract:

The divalent metal complexes of $[Cu(II)(L)(H_2O)_2]$ was obtained by the reaction of new hydrazone Schiff base ligand of heterocyclic 2-hydroxybenzylidene)-4-oxopiperidine-1-carbohydrazide (H_2L). The metal (II) complex was analyzed and characterized by elemental analysis, some routine spectral techniques viz., FT-Infrared, UV-visible spectroscopy, and powder XRD. The stoichiometric ratio of hydrazone ligand and its Cu(II) metal was observed as 1:1 (M:L) in the complex. The spectral, TG data suggested a tetrahedral structure of Cu(II) complex.

KEYWORDS: Hydrazone Schiff base, divalent metal complex, spectral characterization, fluorescence emission, antifungal and anti-bacterial study.

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1. INTRODUCTION:

The development of a Schiff base is a significant stage in numerous biochemical responses. The nitrogen of $>C=N$ group in mix with other contributor bunches present in the atom make Schiff bases great ligands. The Schiff base class is extremely adaptable as mixtures can have a wide range of substituents and they can be unabridged or N,N' -cross over [1-5]. Schiff bases that contain aryl substituents are considerably steadier and all the more promptly orchestrated, while those which contain alkyl substituents are generally unsteady. Schiff bases of aliphatic aldehydes are generally temperamental and promptly polymerizable, while those with fragrant

aldehydes having compelling formation are steadier [6-8]. The development of Schiff base from an aldehyde or ketones is a reversible response and by and large happens under acid or base catalysis, or after warming. As a rule, aldehydes respond quicker than ketones in Schiff base buildup responses as the response focus of aldehyde is sterically less prevented than that of ketone. Moreover, the additional carbon of ketone means electron thickness and hence makes the ketone less electrophilic contrasted with aldehyde [9-12]. In azomethine derivatives, $(>C=N)$ linkage is very important for biological activity, reported remarkable activities as an antibacterial, antifungal, anticancer and diuretic properties **fig.1.and 2.**

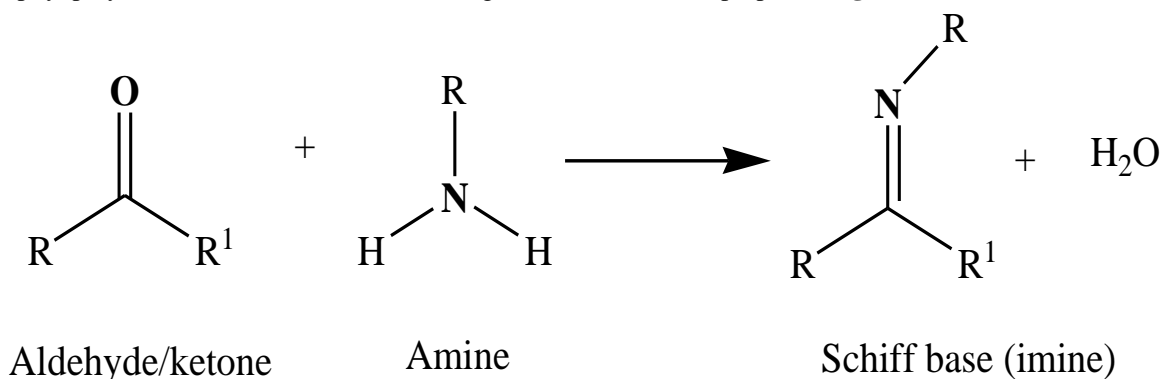


Fig.1 General Synthesis of Schiff base

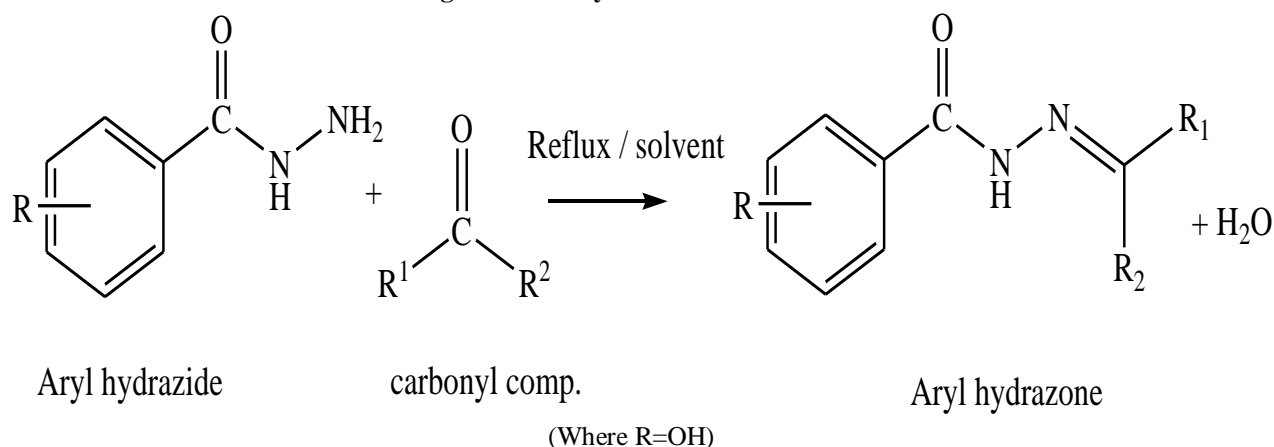


Fig.2 General Synthesis of hydrazone Schiff base ligand

2. EXPERIMENTAL:

2.1 SYNTHESIS OF COPPER (II) COMPLEX:

A hot solution of hydrazone Schiff base of 2-hydroxybenzylidene)-4-oxopiperidine-carbohydrazone (H_2L) in equimolar amounts was reacted to $CuCl_2 \cdot 6H_2O$ to obtained $Cu(II)$ complex. The pH ~ 7.7 of the solution was kept with a mixture of alcoholic ammonia solution (2.5 ml). The product was filtered and washed by warm ethanol. The elemental analysis data, molecular formula, Molecular wt., and its molar conductivity data of metal complexes are reported in (Table 1).

Table1. Analytical data of hydrazone Ligand (H₂L) and its Copper (II) complex

Compounds	M. Formula	Formula wt.	Element found (calcd.) %					Molar cond. (Sm ² mol ⁻¹)
			C	H	N	Cl	M	
Hydrazone Schiff base	C ₁₃ H ₁₅ N ₃ O ₃	261.27	59.90 (59.76)	5.90 (5.78)	16.87 (16.08)	---	----	---
Cu(II) complex	CuC ₁₃ H ₁₈ N ₃ O ₅ Cl	395.29	39.55 (39.49)	4.65 (4.58)	10.85 (10.62)	9.10 (8.96)	16.20 (16.07)	4.12

2.2 MATERIALS:

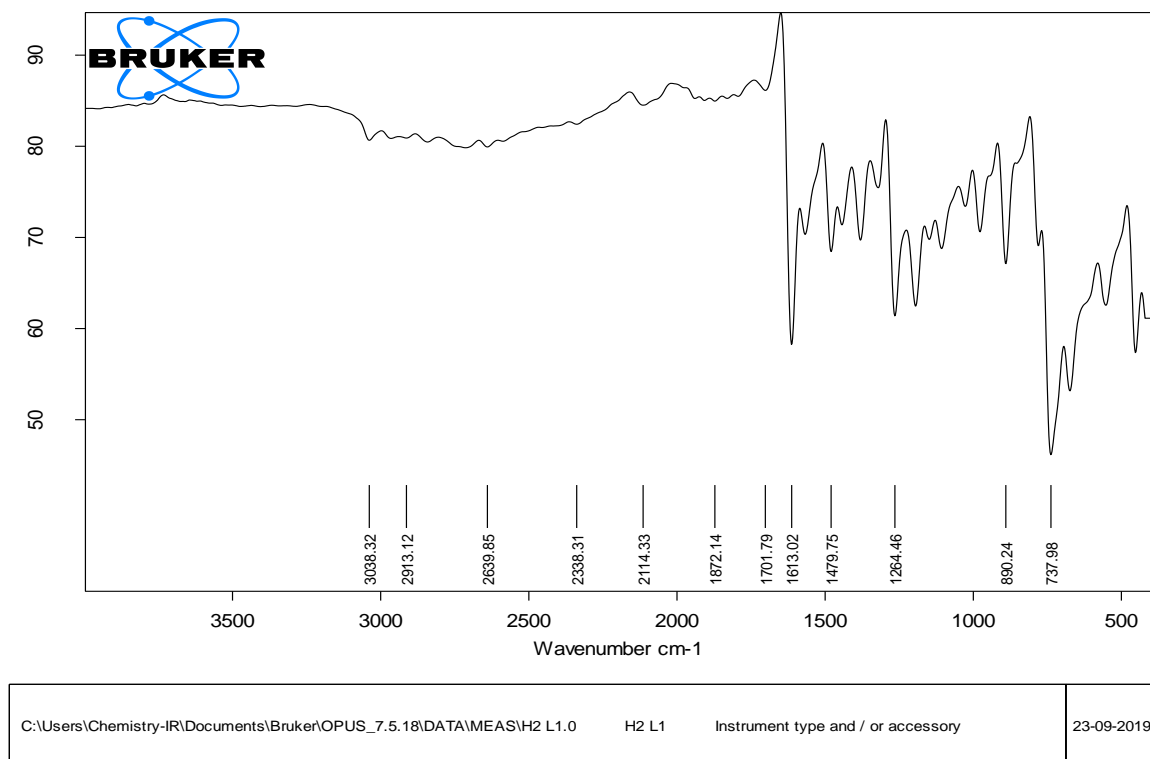
All the chemical were used are of analytical grade from S.D. fine chemicals, and Hi-media. The solvents were purified by the distillation by rota vapours.

2.3 ANALYSIS AND PHYSICAL MEASUREMENTS:

FT-Infrared (IR) spectra was measured on Bruker Advance spectrophotometer. The elemental analysis were carried a on the Carlo-Erba analyzer. A Sherwood magnetic susceptibility balance (MK-1) was used to measure the magnetic susceptibility. PMR and ¹³C-NMR spectra of ligand in DMSO-*d*₆ in (TMS) on a Bruker spectrophotometer. The mass spectrum of (ESI) of Schiff base ligand was measured on micro-mass spectrophotometer. The UV-visible spectrophotometer double beam Shimadzu (1800) was utilized to record absorption spectra. The ESR spectrum of Cu(II) complex was measured on ESR Spectrophotometer (JES - FA200) in liq.N₂. The thermogravimetric analysis were measured on (TGA-4000) Perkin-Elmer analyzer in N₂ atmosphere. Powder X-ray diffraction were recorded on Rigaku-Miniflex (600). Elico conductivity meter-180 was used for the molar conductivities.

3. RESULTS AND DISCUSSION:**3.1 IR spectral characterisation of Cu(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 Fig.3(a) and absorption band recorded in table 2. Cu(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 H₂O 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 [13-15]. On the basis of the IR spectral data of (Table 2) and Figure (1) hydrazone ligand and Fig.(2) Cu(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 shown in Fig.3(b).



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Fig.3 (a) FT-IR spectrum of Ligand

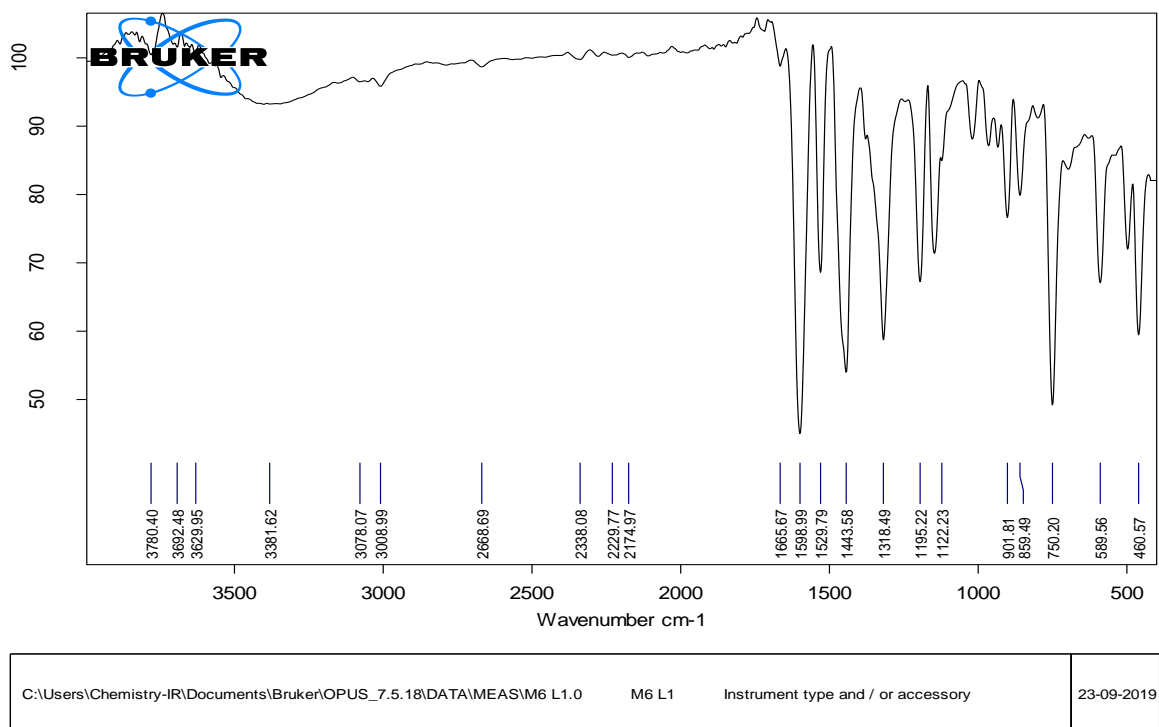


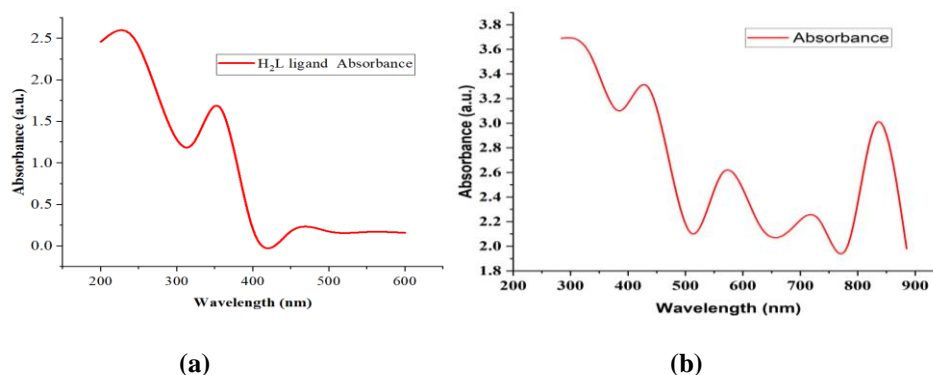
Fig.3 (b) FT-IR spectrum of Cu(II) complex

Table 2. FT-IR spectral data of hydrazone Schiff base (H₂L) and Copper (II) complex (cm⁻¹)

Compound (cm ⁻¹)	$\nu(\text{OH})$	$\nu(\text{N-H})$	$\nu(\text{C=N})$ azom.	$\nu(\text{C-O})$ Phen.	$\nu(\text{C-O})$ enolic	$\nu(\text{N-N})$	$\nu(\text{H}_2\text{O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
Hydrazone ligand	3377	3260	1632	1300	---	951	---	---	----
Copper(II) complex	----	3281	1598	1318	--	980	859	589	460

3.2 Magnetic moment, and UV-Visible spectroscopic studies:

Electronic absorption: The prominent σ , π and lone pair electrons undergoes excitation from the ground to excitation energy state. The azomethine $-\text{N}=\text{C}-\text{H}$ ($\pi \rightarrow \pi^*$) transition shifted to red shift higher wavelength in the region of 405-432 nm shown in Fig. 4.(a) and (b). The band arises due to the transition of nonbonding (lone pair) π electron into antibonding π^* orbital on oxygen atom of carbonyl moiety in wavelength region of 270-300 nm. The Cu(II) complex showed broad band around at 832nm due to ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$ transition in ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$ indicating characteristic of a distorted octahedral copper (II) complexes with an axial elongation. The bands observed at 565 and 434 nm are due to ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$ and ligand to metal charge transfer $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, respectively [24]. Copper (II) complex shows magnetic moment of 1.70 B.M. [16-17].

**Fig.4 Electronic absorption spectra of (a) hydrazone Schiff base ligand (b) Cu(II) complex**

3.3 Emission spectroscopy of ligand and Cu(II) complex:

The hydrazone ligand (H₂L) ligand and its Cu(II) complex was excited at 390nm in slit-2, the fluorescence emission recorded at 558, 537nm shows good emission intensity such as 260.84, 5.88 (a.u.) respectively. The fluorescence emission spectra of (H₂L), and its Cu(II) metal complexes was recorded on JY Fluorolog-3-11 spectrophotometer in both excitation radiation simultaneously emitted radiation. The solid compound scanned in the range of 200-800 nm using energy source Xenon Lamp-450W and resolute spectra at 0.2 nm using software data max/grams/31. The steady state fluorescence spectrum of a compounds consists of collection of emission light in a range of selected wavelength by exciting the compounds at a specific wavelength. The excitation spectrum consists of collection of emission from a sample at a specific wavelength by exciting the sample in a range of wavelengths 250-800nm. The

fluorescence is created by the absorption of light energy by fluorescent molecule called fluorophore. The intensity of the fluorescence signal is usually measured on fluorescence plate readers of the light signal emitted by a sample in relative fluorescent unit. It is more sensitive than UV visible spectroscopy where fluorescence measured directly without any reference beam. Comparatively, hydrazone ligand shows the high intense emission intensity than Cu(II) complex. The emission intensity was due to aromatic nature of compounds, the photo induced electron transfer (PET), fluorophore groups, magnetic perturbation and static interaction of metal ion toward fluorophore group in the structure is due to the charge transfer in ligand and in metal complexes (ligand) $\text{L} \rightarrow \text{M}$ (metal). The ligand and its metal complexes reflected their fluorescence emission spectra and found order of the emission as $\text{H}_2\text{L} > \text{Cu(II)}$ [18-19]. The spectra do not show any new band at higher wavelength region, which indicated the non-formation

of emissive in wavelength region. The photoluminescence i.e. Fluorescence intensity is depend upon the presence on the fluorophoral group and mechanism of metal to ligand charge transfer (MLCT) or ligand to metal charge transfer (LMCT) in metal complexes and plotted in arbitrary unit (a.u.) or count per second (cps). The fluorescence intensity indicates light (photons) is emitted is depend on the

concentration of the excited fluorophore and their structural phenomenon.

This shows that there is no change in the structure and geometry of fluorophore in the excited state. Based on the above emission, it was revealed that the hydrazone ligands and its metal complexes are referred as good fluorescent components and may be used in photochemical applications and in the detection of heavy metal ion biological samples [20-22].

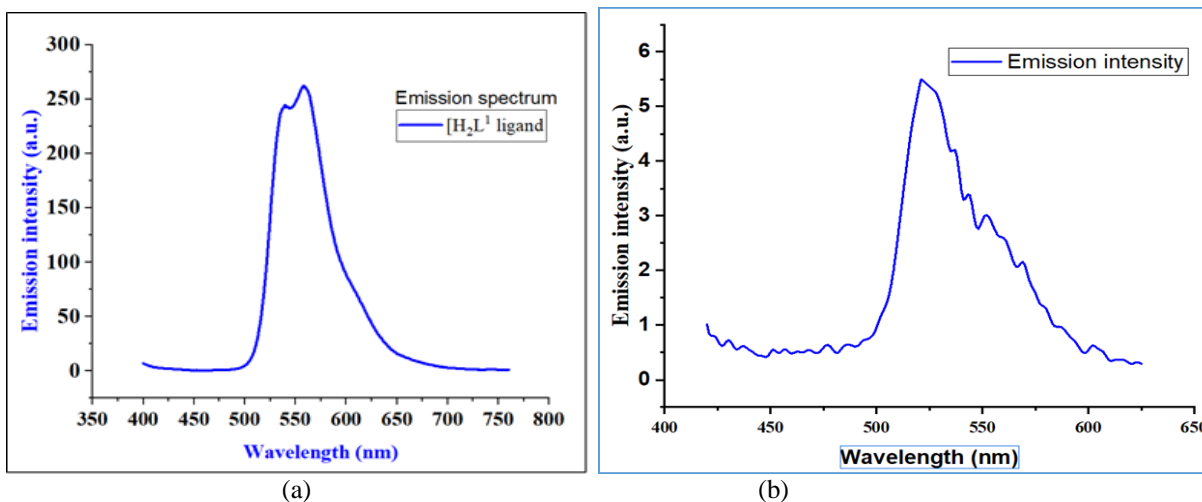


Fig.3 Emission spectra of (a) hydrazone ligand (b) Cu(II) complex

3.4 ESR spectra of Cu(II) complex:

Electron spin resonance spectroscopy is the study of the absorption of the microwave radiation frequency by the paramagnetic substances to induce the transition between magnetic energy levels of electrons with unpaired spins. ESR is a powerful non-destructive analytical technique invented by physicist Zavoiskii in 1944. The magnetic field splitting are occurred by applying a static magnetic field. The resonance is occurs due to spin of odd or unpaired electron present in the partly filled orbital. ESR spectra of all five Cu(II) complexes were recorded on JES-FA200 ESR Spectrophotometer with X band at E standard frequency (X band) range of 8.75-9.65GHz frequency at temperature 77k in liquid nitrogen. The X-band spectrum of Cu(II) complex consist of one g_{\parallel} and g_{\perp} peak at 305.39 mT, 343 mt respectively and its spectrum is presented in Fig.4. The spectrum was observed g_{\parallel} 2.153 and g_{\perp} 2.055 which indicating axial symmetry and shows electron predominantly in $d_{x^2-y^2}$ orbital. The molecular orbital coefficients correlation was derived from α^2 -(covalency of in σ bonding) and β^2 -(covalency of in π bonding). The equation of $\alpha^2 = (A_{\parallel} / 0.036) + (g_{\parallel} - 2.0023) + 3/7(g_{\perp} - 2.0023) + 0.04$, $\beta^2 = (g_{\parallel} - 2.0023) E / 8\lambda\alpha^2$. The result obtained β^2 (0.76) > α^2 (0.69) inferred that covalency of in π bonding was stronger than covalency of in σ bonding [23-24].

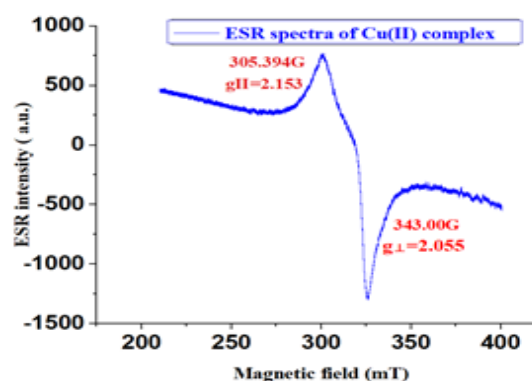


Fig.4 ESR spectrum of Cu(II) complex

3.5 Thermal study of Copper (II) complex:

The aim of the present investigation was to study the thermal proprieties of synthesized hydrazone ligand and its copper (II) complex and its thermogram of hydrazone ligand and (Cu(II) complex represnted in Fig.5. (a) and (b). The Thermal curve of ligand (H_2L) shows two steps of decomposition in the temperature range 45-647 °C [25-26]. The thermogram was almost stable up to 200 °C, and did not shown any weight loss and with further subsequent heating shows a broad (DTG_{max}) peak in between 350-370 °C corresponding

to mass loss of 90.12 % (Calcd.90%) due to the elimination of organic moiety, and finally curve attain a horizontal level at 647 °C leaving as carbon residue 14.2 % (Calcd.14 %). TG curve of $[\text{Cu}(\text{HL})(\text{H}_2\text{O})_2\text{Cl}]$ complex shows mass loss at 120 °C with a mass loss of 10.0 % (calcd. 9.1%) are due to the loss of two coordinated water molecule, in second degradation

stage occurred at 210 °C (DTG_{max}) with a weight loss of 10.0 % (calcd. 9.9%) due to the loss of one chloride ion. In third stage decomposition observed at 450°C with a mass loss of 70.8% (calcd. 70.0 %) which corresponds to the loss of remaining organic moiety of ligand and finally the complex is converted into its stable metal oxide 11.0% (calcd.10.6 %) [27-28].

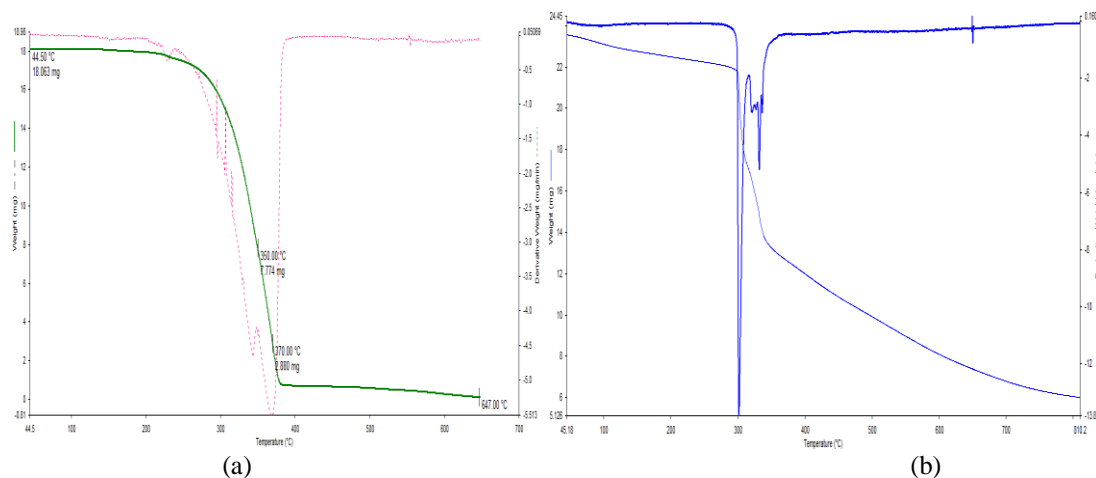


Fig.5 Thermogram of (a) hydrazone ligand and (b) Cu(II) complex.

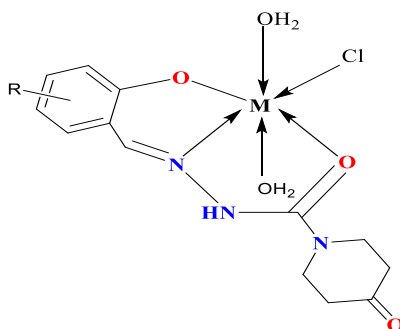


Fig.6. Proposed Octahedral structure of Cu(II) complex (Where M=Cu(II)ion.

CONCLUSION:

The mononuclear complex of the type $[\text{CuC}_{13}\text{H}_{18}\text{N}_3\text{O}_5\text{Cl}]$ have been characterized by spectro-analytical techniques. The molar conductance parameters inferred that the complexes are non-electrolytic behaviors. Based on the above emission, it was revealed that the hydrazone ligands and its metal complexes are referred as good fluorescent components and may be used in photochemical applications and in the detection of heavy metal ion biological samples.

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