

# Synthesis and Characterization of TosylHydrazone Complexes Derived from TosylHydrazone

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**Abstract**—A new Schiff base ligand and complexes have been synthesized from tosylhydrazone 1,10phenanthroline and metal chloride salt of Ba(II), and Zinc(II) in acetonitrile medium. Several methods were used in particular; CHNS analysis, <sup>1</sup>H NMR, IR, UV-Vis, Molar conductance, Magnetic moment and mass spectra. On the basis of elemental analysis and spectral examination, five corresponding geometries were given to these two complexes. Based on these results, it is observed that this ligand acts as a bidentate and coordinates through oxygen and nitrogen atoms of the ligand. And 1,10phenanthroline binds through two symmetrical nitrogen atoms. Complexes are non-hygroscopic and photo stable solids at room temperature.

**Index Terms**— Ligand, metal complex, photo stable., Schiff base, Spectral studies.

## I. INTRODUCTION

Schiff bases are acquired from an amino and carbonyl compound are a significant class of ligands that arrange to metal particles by means of azomethine nitrogen and have been examined widely. In azomethine subordinates, the C=N linkage is fundamental for natural movement, a few azomethine mixes have been accounted for to have striking anticancer and antimalarial antibacterial, antifungal, exercises. The tosylhydrazone ligand and can be utilized for combination of an assortments of mixes, with 1,10 phenanthroline, and metal salts. Azo Schiff base metal edifices have been examined widely for a considerable length of time because of the manufactured adaptabilities of these Schiff base ligand and their affectability just as selectivity towards the progress metals [1]. Amongst the ligand frameworks, hydrazones possess uncommon spot since progress metal edifices of these ligand created because of their chelating capacity, basic adaptability, fascinating electrical just as attractive possessions [2, 3], and, these days, these are broadly existence utilized on behalf of their auspicious applications in handling of a few illnesses and furthermore existed utilized as manufactured and systematic substances [4], as polymer-covering shade and fluorescent materials [5–9].

**Revised Version Manuscript Received on 16 September, 2019.**

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Besides, these ligands display keto-enol tautomerism [10–14] demeanor surprising synchronization numbers. In the present paper, reported the union and portrayal of Ba(II), and Zn(II) edifices of Schiff base got from the buildup of toluene sulphonylhydrazide and 1,3 Diphenyl acetone respectively. The methylol derivatives of 2,6-dimethylol-4-bromophenol were synthesized by the condensation of 4-bromophenol with formaldehyde within the sight of NaOH by way of a catalyst in very critical conditions.

## II. EXPERIMENTAL

Toluene sulphonylhydrazide, metal chloride salts and 1,3 Diphenyl acetone were procured from Sigma-Aldrich and utilized directly as acknowledged. All solvents were of reagent grade and were utilized moving forward without any more filtration. Make note IR spectra such as KBr pellets over a Bruker FTIR 1003-3610 Alpha-P spectrometer. NMR measurements were performed on a Bruker 400 MHz Multinuclear spectrometer. The samples are broken up in d<sub>6</sub>-DMSO with TMS as inside reference. The magnetic susceptibility estimations of the paramagnetic complexes were done on a Sherwood Scientific magnetic vulnerability balance. The electronic retention spectra were estimated over a Perkin Elmer Lambda 650 UV-Vis spectrophotometer. The complexes were additionally portrayed by essential investigation Model: LECO Tru Spec CHN(S). CHNS ANALYSER

## III. SYNTHESIS OF SCHIFF BASE

Fig.1 Synthesis of TosylHydrazone



It is synthesized by refluxing of toluene sulphonylhydrazide (0.01 M) and 1,3Diphenyl acetone (0.01M) in methanol, for 4-5 hours within the sight of a 2-3 drops of glacial acetic acid. The response mixture was cooled to ice temperature, wherein whitish-yellow color substance attained. It is purified by washing several times using methanol and dried up in vacuum desiccators'. The pure substance was re-crystallized as of methanol.

3.1 Synthesis of metal complexes

0.01 M of Tosylhydrazone solution (10ml) and 0.01 M metal chloride salt was mixed with acetonitrile solution (10ml) in round bottom flask. The reaction mixwas refluxed for 5 to 6 hours. then 1,10 phenanthroline was added and heated for 8-10 hours. Pale yellow color complex was allowed to digest and collected by filtration over filter paper.

Fig.2 Synthesis of Zn complex

TABLE.1 elemental analytical data (%) and m/z value of L and complexes.

compounds	Elemental (%)				Color	m/z
	C	H	N	S		
Ligand	69.71	5.36	7.30	8.17	yellow	378.14
	69.81	5.86	7.40	8.47		
[ZnL(Phen)Cl]	61.02	4.15	8.18	4.25	Ivory	658.11
	61.82	4.75	8.48	4.85		
[BaL(Phen)Cl]	55.05	4.13	7.25	4.07	White	732.09
	55.75	4.27	7.65	4.37		

Calculated and observed values are given in the table.

Fig.3 Synthesis of Ba complex

IR spectra

The band stretching vibration frequency at 2400cm<sup>-1</sup> in ligand which is shifted to lower wave number in complexes. [13]. Besides, ligand exhibits stretching of (NH) at 3300cm<sup>-1</sup> and (C=N) stretching frequency at 1590 cm<sup>-1</sup> which on complexation shifted to lower frequency regions, suggesting sulphonyl, and nitrogen groups are coordinated to the metal

atom. Similarly, a band (N-H) stretching frequency at 3300 cm<sup>-1</sup> in ligand remains unaffected in complex suggesting evidence for its non-coordination. A new band at 3250cm<sup>-1</sup>, 3500 cm<sup>-1</sup>, 3100cm<sup>-1</sup> in Ba(II) [16], Zn(II) are due to coordination of 1,10 phenanthroline.[17-18] and due to the presence of coordinated groups. The appearance of new bands in the spectra of Zn(II) and Ba(II) metal complexes at 500 ,490cm<sup>-1</sup> due to (M-N) [19-20]. Also, Metal-oxygen bonding (M-O) is at 600 and 700 cm<sup>-1</sup> [21-22].

IV. RESULTS AND DISCUSSION

Ligand is Ivory colored and complexes are colorless and steady at room temperature. These are unsolvable in water and solvable in diluents like DMF, DMSO.

4.1 Characterization of the prepared ligand, and complexes, study of IR Spectra

After preparation of metal complexes, color, decomposition point, yield physical characterization similar to nature, magnetic moment (B.M.) of the complexes was dignified. The molar conductance values, Conductivity in DMF disclose their non-electrolytic nature[9].

TABLE 2.IR spectral data of Ligand and complexes.

Compounds	Wavelength (cm <sup>-1</sup> )			
	ν (C≡N)	ν SO	M-O	M-N
L	1590	2400	-	-
[ZnL(Phen)Cl]	1600	2430	600	490
[BaL(Phen)Cl]	1650	2450	500	700

TABLE 3.NMR spectral data of Ligand and complexes.in ppm.

Compounds	-CH-	Aromatic	NH
HL	2.4	7-7.5	--
ZnL(Phen)Cl	2.3	7--8	8.9
BaL(Phen)Cl	2.3	7--8	8.9

4.2 Conductivity and Magnetism;

Ligand and complexes are non-electrolytes with low conductivity values around 10-25 ohm<sup>-1</sup> cm.<sup>2</sup>They are diamagnetic with very low magnetic moment values. UV-Vis and magnetic moment indicate that both complexes have square pyramidal geometry.

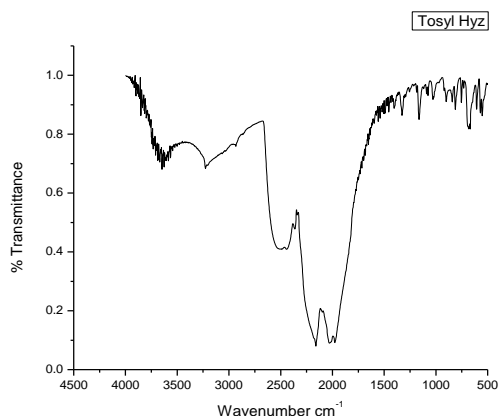


Fig. 4 IR spectrum of Ligand

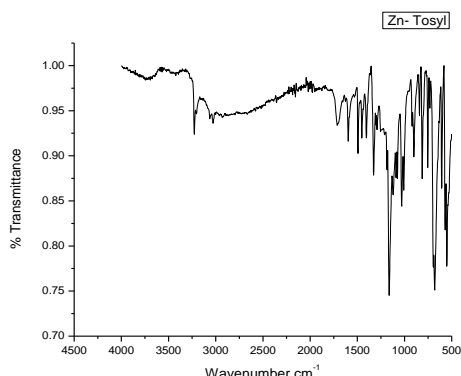


Fig. 5 IR spectra of Zn complex

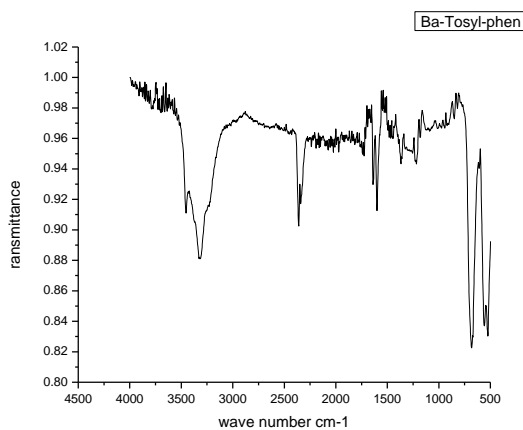


Fig. 6 IR spectra of Ba complex

#### 4.3 Study of <sup>1</sup>H NMR spectral analysis of ligand, Zn and Ba complex.

<sup>1</sup>H NMR signals at 7-8 ppm correspond to aromatic protons and at 9.0ppm because of NH proton. A resilient signal at 9.0ppm is assignable to NH protons in complexes. Signal of NH group shifted to up field region in Zn(II) and Ba(II) metal complexes. In metal complexes signal conforming to 1,10phenanthroline group has appeared. A prominent peak due to the presence of CH<sub>2</sub> at 2.3 ppm is observed in both the complexes. Thus, ligand molecule is by all accounts composed to the metal azomethinenitrogen (C=N) in Zn(II) and Ba(II) complexes.

Fig. 7 NMR spectrum of Ligand

Fig. 8 NMR spectrum of Zn complex

Fig. 9 NMR spectrum of Ba complex

Table 4. Electronic spectral data of Ligand and complexes indmso.

Compound s	Absorption bands (cm <sup>-1</sup> )	Assigned transition
Ligand	460	$\pi \rightarrow \pi^*, \pi \rightarrow \pi^* n \rightarrow \pi$
[ZnL(Phen) Cl]	550	$\pi \rightarrow \pi^*, n \rightarrow \pi^*$ ,
[BaL(Phen) Cl]	580	$\pi \rightarrow \pi^*, n \rightarrow \pi^*$

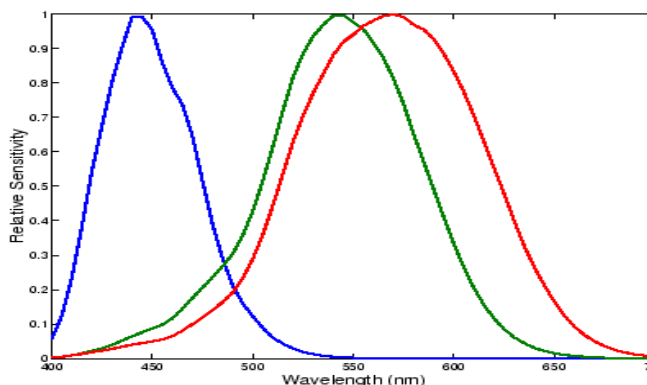


Fig. 10. UV-Vis spectrum of Ligand and complexes

*Biological studies;*

The results depicted that the methods are valid for finding the MIC, the MBC, and for performing the antimicrobial susceptibility test through disk diffusion method. The results demonstrate that the appropriateness of the antimicrobial measures with plant extracts aimed at the ligand and complexes. The data obtained in the bio-assays are quantitative naturally, and are identified with the weakness of the bacterial strains towards the tested compounds. The Microbial strain on gram-positive and gram-negative bacteria exhibited high susceptibility to the tested compounds,. Besides, the MIC values acquired for these metal complexes showed their impending for pharmacological usage. In any case, further investigations with respect to the development restraint component and toxicity are observed to be of fundamental significance so as to assess the capability of these mixes in therapeutic application.[23-26] Moreover, the results of analysis presented a fair antimicrobial action of the ligand and metal complexes. However complexes have better ability than their ligands

**V.CONCLUSION**

The ligand 1,3 diphenyltosylhydrazone Zinc(II) and Barium(II) complexes were successfully synthesized The ligand, 1,3 diphenyltosylhydrazone was synchronized towards two dissimilar metal ions through oxygen and nitrogen atoms. To get the conforming complexes. The complexes are five-coordinated and exhibited square pyramidal geometry. Preliminary *in vivo* antibacterial revision designated that all the complexes attained exhibited a modest activity in contrast to the tested bacterial strains and a somewhat higher activity compared to the ligand, (L). Based on spectral and analytical data of Zn(II) and Ba(II) complex, synthesis of two complexes has been confirmed.

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