

## Synthesis and Characterization of 5-Substituted-(2-methylbenzimidazol-1-yl)-1,3,4-oxadiazole-2-thione and Its Metal Complexes of Fe(III), Hg(II), Zn(II) and Sn(II)

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**Abstract:** A ligand 5-Substituted-(2-methylbenzimidazol-1-yl)-1,3,4-oxadiazole-2-thione (L) and its Fe(III),Hg(II),Sn(II) and Zn(II) complexes were synthesized. The authenticity of the ligand and its transition metal complexes were established by elemental analyses, conductance and magnetic susceptibility measurements, as well as spectroscopic (IR,  $^1\text{H}$  NMR, UV-Vis and Mass) and thermal studies. All complexes exhibit the composition  $\text{ML}_2\text{X}_2$  where M =Fe(III),Hg(II), Zn(II) and Sn(II), L=Ligand, X=OAc and chloride ions. The IR spectral studies revealed the existence thiol-thione tautomerism in the ligand molecule. The ligand acts as a bidentate coordinating through the N-3 nitrogen and the exocyclic sulfur atoms of ox diazole rings. Antimicrobial screening of the ligand and its metal complexes were determined against the bacteria *Escherichia coli* and *Salmonella paratyphi A*.

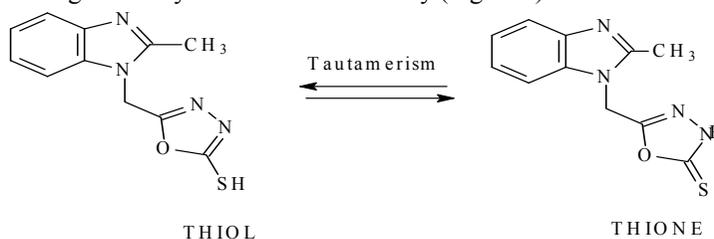
**Keywords:** Oxadiazoles, Benzimidazoles, Transition metal complexes, Electronic spectra, IR spectra, Biological activity

### Introduction

Benzimidazole derivatives play vital role in biological field such as antimicrobial, antiviral, anti-diabetic and anticancer activity<sup>1</sup>. Therapeutic significance of these clinically useful drugs in treatment of microbial infections encouraged the development of some more potent and significant compounds. Benzimidazoles are remarkably effective compounds both with respect to their bacteria inhibitory activity and their favorable selectivity ratio<sup>2</sup>. Benzimidazoles are regarded as a promising class of bioactive heterocyclic compounds that exhibit a range of biological activities<sup>2</sup>. Specifically, this nucleus is a constituent<sup>2</sup> of vitamin-B<sub>12</sub>.

On the other hand 1,3,4-Oxadiazoles are of considerable pharmaceutical and material interest<sup>3-5</sup>.They have been shown to possess muscle relaxant, anti mitotic, analgesic, anti-

inflammatory, anticonvulsive, diuretic and anti-emetic properties<sup>3-5</sup>. They also possess tranquilizing, anti-tubercular, hypoglycemic, herbicidal, antiviral, amoebicidal, insecticidal, hypnotic and sedative activities. Some material applications of 1,3,4-oxadiazole derivatives lie in the field of liquid crystals<sup>5</sup>. Oxadiazole derivatives, which belong to an important group of heterocyclic compounds, have been the subject of extensive study in the recent past. Numerous reports have highlighted their chemistry and use<sup>3-5</sup>. Diverse biological activities, such as anti-tuberculostatic, anti-inflammatory, analgesic, antipyretic and anticonvulsant<sup>4</sup>, have been found to be associated with oxadiazole derivatives<sup>5</sup>. For this reason our aim was to synthesize various 1,3,4-oxadiazole-2-thione derivatives to make notable contributions to this class of heterocyclic compounds. We report the synthesis and characterization of some 5- substituted-1,3,4-oxadiazole-2-thiones using the synthetic procedure based on the ring closure reactions of appropriate acid hydrazides with carbon disulphide. The heterocyclic thiones represent an important type of compound in the field of coordination chemistry because of their potential multifunctional donor sites, *viz* either exocyclic sulfur or endocyclic nitrogen<sup>6-7</sup>. There are a few reports on the metal complexes of 5-substituted-1,3,4-oxadiazole-2-thione<sup>4</sup>. To the best of our knowledge we are reporting for the first time metal complexes based on 1,3,4-oxadiazole-2-thione containing 2-methyl-benzimidazole moiety (Figure 1).



**Figure 1.** Proposed structure of the title ligand

## Experimental

Commercial reagents were used without further purification and all experiments were carried out in open atmosphere. All the solvents were purchased from Merck Chemicals, India, and used after purification.

Carbon, hydrogen and nitrogen contents were estimated on a Truspec model micro-analyser. By following standard procedures<sup>8</sup>, the complexes were analyzed for their metal content. For metal ion determination, a solution of the complex was first prepared by decomposing a known amount of complex with a mixture of conc. nitric acid and conc. sulfuric acid<sup>8-9</sup>. The digestion was continued with repeated addition of acid mixture to ensure complete decomposition of organic matter in the complex. The residue was extracted with distilled water and the metal contents were determined by standard methods. The sulfur was estimated as BaSO<sub>4</sub> by gravimetric method<sup>8</sup> and chloride was estimated by volumetric method<sup>8</sup> using standard AgNO<sub>3</sub> solution. The molar conductance measurements were made in DMSO with 10<sup>-3</sup> M solutions<sup>9</sup> using an EI Conductivity meter, Type CC-01 with a cell constant of 1.02.

Magnetic susceptibility measurements were performed at room temperature on a Gouy balance using Hg[Co(NCS)<sub>4</sub>] as the calibrant<sup>9</sup>. IR spectra were recorded in the 4,000–400 cm<sup>-1</sup> region (for the ligands) IR Spectrophotometer PE FTIR and in 4000-200 cm<sup>-1</sup> region (for metal complexes) on Nicolet instrument model MAGNA 550 spectrophotometer as KBr pellets<sup>11</sup>. <sup>1</sup>H NMR spectra were recorded in DMSO-d<sub>6</sub> on Bruker AV III NMR spectrometer using TMS as internal reference. The EI- Mass spectrum was recorded on JEOL GC -MATE GC--MS Spectrometer using 70 eV with a source temperature of 150 °C. The thermal studies of the

complexes were performed on a NETZSCH TGA/DTA/DSC Thermal analyzer under a nitrogen atmosphere at a heating rate of 10 °K/min in the temperature range of 50 to 1400 °C.

### Synthesis of ligand

Following steps are involved in the synthesis of ligand. The reactions involved in the synthesis of the ligand are shown in Scheme 1.

#### Preparation of 2-methylbenzimidazole<sup>1</sup>

A mixture of *o*-phenylenediamine (10 g, 0.1 mole) & acetic acid (8.4 mL, 0.14 mole) was heated under reflux for 2 h. Then the reaction mixture was cooled & it was made alkaline by addition of sodium bicarbonate. The solid thus separated was collected by filtration and dried at 100 °C. Yield, 6.5 g (60%) mp, 172-174 °C.

#### Synthesis of (2-Methylbenzimidazol-1-yl)-acetic acid ester<sup>2</sup>

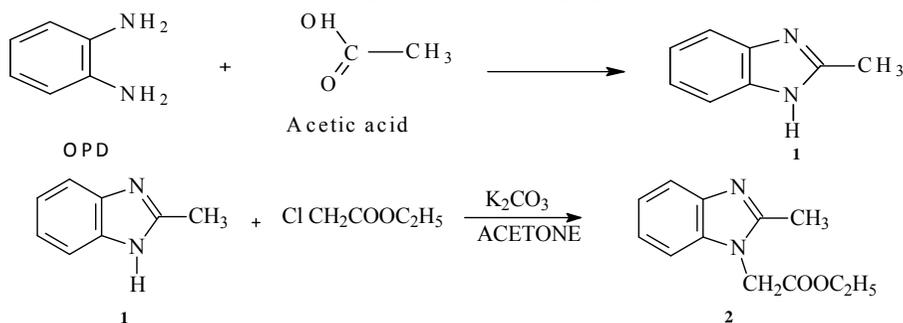
A mixture of 2-methylbenzimidazole(1) (6.5 g, 0.05 mole), Ethyl chloroacetate (10.0 mL, 0.075 mole) and potassium carbonate(15 g) in acetone(100 mL) as a solvent was refluxed for 6 h. The reaction mixture was cooled to room temp and acetone was removed by distillation. Then the reaction mixture was transferred to ice cold water and it was stirred to dissolve the potassium carbonate. The solid ester thus separated was filtered, washed with water, dried and recrystallized with ethanol. Yield, 6.0g (60%) mp, 82-84 °C.

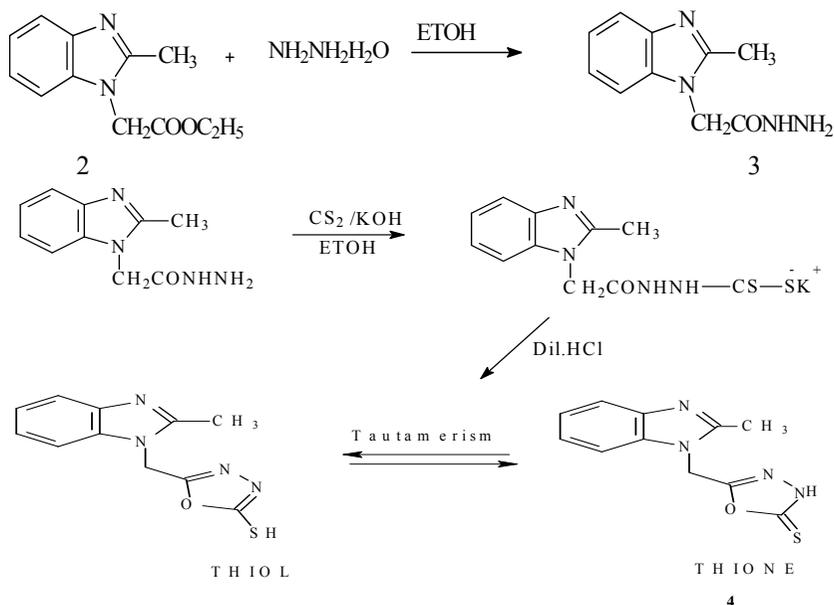
#### Synthesis of (2-Methylbenzimidazol-1-yl)-acetic acid hydrazide<sup>2</sup>

A mixture of (2-Methylbenzimidazol-1-yl) acetic acid ester(2) (5 g, 0.023 mole) and hydrazine hydrate(3.0 mL, 0.058 mole, 2.5eqv) in ethanol(25 mL) as a solvent was refluxed for 4 h. The solution on cooling gave solid mass of hydrazide. The solid was recrystallized with ethyl alcohol. Yield, 4.5g (90%) mp, 210-212 °C.

#### Synthesis of 5-Substituted-(2-Methylbenzimidazol-1-yl)-1,3,4-oxadiazole-2-thione<sup>2</sup> (MBIMOT)

To a solution of potassium hydroxide (1.7 g) in ethanol (25 mL) (2-methyl benzimidazol-1-yl)-acetic acid hydrazide (3) (4.0 g, 0.02 mole), was added followed by carbon disulfide (3.0 mL, 0.04 mole). The reaction mixture was heated under reflux for 6 h. Then the reaction mixture was allowed to cooled to room temp. Then it was concentrated, diluted with minimum amount of distilled water and acidified with 1 N HCl. The solid thus separated was collected by filtration. It was then washed with water dried and recrystallized with a mixture of methanol and DMF. The Scheme 1 shows the steps involved in the preparation of legand(4) MBIMOT.





**Scheme 1.** Reactions involved in the synthesis of ligand

*General procedure for preparation of metal complexes using MBIMOT as a ligand<sup>10-11</sup>*

In the preparation of metal complexes of various transition metal ions using MBIMOT as a ligand a general procedure was followed<sup>10-11</sup>. The ligand and metal salts were taken in 2:1 ratio<sup>10</sup>. The ligand was dissolved in DMF and the hot solution of ligand was mixed with a hot aqueous solution of various metal salts. The complexes were precipitated immediately. In some cases the precipitation occurred when pH of the solution was raised by the addition of sodium acetate. The precipitated complexes were allowed to stand for 1h then filtered, washed with water and ether. Then dried and cooled in a desiccators. The analytical data of ligand and metal complexes is included in Table 1.

**Table 1.** Analytical data of MBIMOT(L) and its metal complexes

Compounds (color)	Mol .wt.	Found (Calculated)%					
		M	C	H	N	S	Cl
$\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS}$ Ligand (Light Yellow)	246	-	53.60 (53.65)	4.00 (4.09)	22.65 (22.75)	12.90 (12.02)	-
$[\text{Fe}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]\text{Cl}$ (Red)	654	8.45 (8.53)	42.5 (42.0)	3.91 (3.80)	15.2 (16.0)	9.65 (9.78)	16.00 (16.28)
$[\text{Hg}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]$ (Yellow)	764	25.03 (25.0)	33.04 (32.90)	3.30 (3.00)	13.9 (14.0)	8.2 (8.0)	9.00 (8.80)
$[\text{Sn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2] \cdot 2\text{H}_2\text{O}$ (Light Yellow)	717	16.40 (16.5)	36.70 (36.82)	3.25 (3.34)	15.25 (15.62)	8.70 (8.92)	9.80 (9.90)
$[\text{Zn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2(\text{OAc})_2] \cdot 3\text{H}_2\text{O}$ (Yellow)	730	8.45 (8.90)	36.00 (36.16)	4.08 (4.38)	16.6 (15.9)	8.5 (8.76)	-

The molar conductivity data obtained in DMSO suggest that the complex  $[\text{Fe}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]\text{Cl}$  is 1:1 electrolyte and other three complexes are non-electrolytes in nature. The magnetic susceptibility properties of the complexes shows that the complex  $[\text{Fe}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]\text{Cl}$  is paramagnetic and three complexes are diamagnetic. The molar conductance values and magnetic susceptibility data are included in Table 2

**Table 2.** Melting point, Molar conductance and magnetic properties of metal complexes

Compounds (color)	mp $^{\circ}\text{C}$	Molar Cond. $\text{Scm}^2 \text{mol}^{-1}$	Magnetic moment. $\mu_{\text{eff}}/\mu_{\text{B}}$
$\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS}$ Ligand (Light Yellow)	275-280 $^{\circ}\text{C}$		
$[\text{Fe}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]\text{Cl}$ (Red)	260-265 $^{\circ}\text{C}$	45.5	4.75
$[\text{Hg}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]$	275-280 $^{\circ}\text{C}$	20.6	Diamagnetic
$[\text{Sn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2].2\text{H}_2\text{O}$	>300 $^{\circ}\text{C}$	25.6	Diamagnetic
$[\text{Zn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2(\text{OAc})_2].3\text{H}_2\text{O}$	295-300 $^{\circ}\text{C}$	18.5	Diamagnetic

### IR spectra

The IR spectra of ligand and complexes have been recorded and the probable assignments are given in the Table 3. The IR spectra of the complexes indicate that the ligand behaves as a bidentate and co-ordinate to the metal via N-3(C-NH) and thiocarbonyl group(C=S) of ox diazole moiety. In the ligand, band appearing at  $3456 \text{ cm}^{-1}$  due to NH stretching is shifted to lower wave number at  $3456 \text{ cm}^{-1}$  -  $3392 \text{ cm}^{-1}$  in the complexes there by confirming the coordination through the nitrogen atom at position 3 of ox diazole ring. The band due to  $\nu \text{C}=\text{S}$  in the ligand at  $1198 \text{ cm}^{-1}$  is shifted to lower wave number at  $1198$ - $1128 \text{ cm}^{-1}$  in the complexes. The shifting of C=S stretching vibration to the lower wave number side as compared to the free ligand is indicative of participation of exocyclic S-atom at C-2 of ox diazole ring in coordination. New bands in the complexes in the region  $595$ - $560 \text{ cm}^{-1}$  compared with IR spectra of free ligand have tentatively been assigned to M-N frequency<sup>11</sup>.

**Table 3.** Major IR bands of ligand(MBIMOT) and its metal complexes.

Compound	N-H	C-H <sup>1</sup>	C-H <sup>2</sup>	C=N	C-O-C	C=S	N-N	M-N	M-OM	OM-Cl	M-S
MBIMOT (L)	3456	3107	2926	1516	1464	1198	926	-	-	-	-
$[\text{Fe}(\text{III})\text{L}_2\text{Cl}_2].\text{Cl}$	3418	3057	2925	1538	1464	1128	938	573	420	361	344
$[\text{Hg}(\text{II})\text{L}_2\text{Cl}_2]$	3443	3056	2924	1585	1451	1138	944	595	420	360	345
$[\text{Sn}(\text{II})\text{L}_2\text{Cl}_2].2\text{H}_2\text{O}$	3392	3053	2926	1525	1465	1128	926	560	420	355	350
$[\text{Zn}(\text{II})\text{L}_2(\text{OAc})_2].3\text{H}_2\text{O}$	3429	3056	2924	1585	1451	1138	976	595	420	360	345

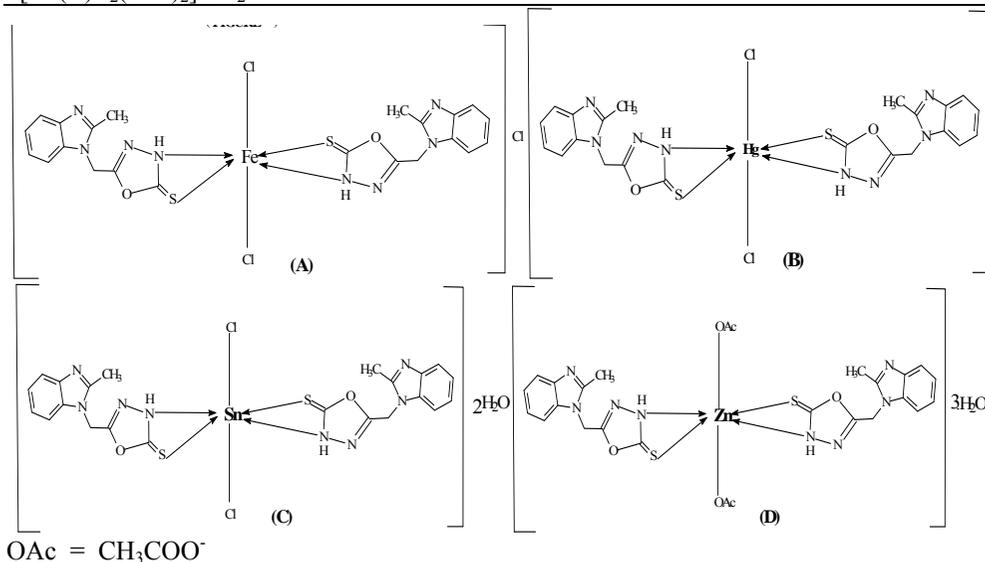
1=Aromatic, 2=Alkyl

### <sup>1</sup>H NMR spectra

The <sup>1</sup>H NMR spectra were recorded and probable assignments are tabulated in Table 4. The <sup>1</sup>H NMR spectra of the ligand have the expected characteristic signals<sup>12-13</sup>. The CH<sub>3</sub> proton shows singlet at  $\delta$  2.60 and N-CH<sub>2</sub> proton at  $\delta$  5.68. In addition doublet peaks at  $\delta$  7.19 and 7.50 may be due to aromatic protons of benzimidazole moiety and a peak at  $\delta$  10.62 may be due to NH proton of ox diazole ring. This confirms the existence of ligand in thione form in liquid state. Signals observed in the complexes at region of  $\delta$  10.59-10.50<sup>13</sup> due to the NH proton are either remained unaffected or shifted slightly downfield with reference to those of the parent ligand and the signal due to CH<sub>3</sub> proton have shown downfield shift in the complexes<sup>14</sup>. The N-CH<sub>2</sub> protons and aromatic protons have shown up field shifts in the complexes. In the complex  $[\text{Zn}(\text{II})\text{L}_2(\text{OAc})_2].3\text{H}_2\text{O}$  an additional peak at  $\delta$  1.93 can be assigned for coordinated acetate(-OOCCH<sub>3</sub>) group protons. These observations support the assigned structure to the complexes (Figure 2).

**Table 4.**  $^1\text{H}$  NMR spectra of ligand (MBIMOT) and its metal complexes<sup>13-14</sup>

Compound	(3H,-CH <sub>3</sub> )	(2H,N-CH <sub>2</sub> )	(2H,Ar)	(2H,Ar)	(1H, NH)	(6H,OAc)
MBIMOT (L)	2.60	5.68	7.19	7.50	10.62	-
[Fe(III)L <sub>2</sub> Cl <sub>2</sub> ].Cl	2.58	5.68	7.23	7.58	10.50	-
[Hg(II)L <sub>2</sub> Cl <sub>2</sub> ]	2.50	6.01	7.58	7.95	10.58	-
[Sn(II)L <sub>2</sub> Cl <sub>2</sub> ].2H <sub>2</sub> O	2.55	5.66	7.17	7.52	10.56	-
[Zn(II)L <sub>2</sub> (OAc) <sub>2</sub> ].3H <sub>2</sub> O	2.50	6.01	7.58	7.95	10.59	1.93

**Figure 2.** Assigned structure to the complexes

### EI-mass spectral studies

The EI-Mass spectrum of the ligand (MBIMOT) showed a molecular ion peak at  $m/z$  246 which is equivalent to its molecular weight. The base peak at  $m/z$  203 is due to the fragment  $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}$ . The fragmentation peaks at  $m/z$  131 and 73 are ascribed due to the cleavage of  $\text{C}_8\text{H}_7\text{N}_2$  and  $\text{C}_2\text{H}_3\text{N}_2\text{O}$  respectively.

The EI-Mass spectrum of the complexes  $[\text{Fe}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]\text{Cl}$ ,  $[\text{Hg}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2]$ ,  $[\text{Sn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{OS})_2\text{Cl}_2].2\text{H}_2\text{O}$  and  $[\text{Zn}(\text{C}_{11}\text{H}_{10}\text{N}_4\text{O})_2(\text{OAc})_2].3\text{H}_2\text{O}$  showed a molecular ion peak  $\text{M}^+$  at  $m/z$  654, 764, 717 and 730 respectively which is equivalent to molecular weight of the complexes. The complex of Fe(III) has base peak at  $m/z$  246 which is due to the loss of a molecule of ligand. The complexes of Hg(II), Sn(II) and Zn(II) have shown the base at  $m/z$  203 which is due to the fragmentation of ligand molecule into  $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}$  in these complexes.

### ESR spectra

The ESR spectrum of Fe(III) complex  $[\text{Fe}(\text{III})\text{L}_2\text{Cl}_2].\text{Cl}$  at RT exhibits three different  $g$ -values indicating magnetic anisotropy in the complex. The distorted structure of the complex is supported by its ESR spectrum recorded in solid state both RT and LNT, which shows anisotropic effects with  $g$ -tensor as  $g_1$ ,  $g_2$  and  $g_3$  respectively at 2.2222, 2.1345 and 2.0341 in consistent with earlier report<sup>14</sup>. The trend  $g_1(2.30) > g_2(2.27) > g_3(2.05)$ , for the complex suggest a rhombic octahedral geometry for the complex.

### Thermogravimetric analysis

The heating rates were suitably controlled at  $10\text{ }^{\circ}\text{C min}^{-1}$  under nitrogen atmosphere and the weight loss was measured from the ambient temperature up to  $1400\text{ }^{\circ}\text{C}$ . The thermogravimetric studies reveal that no water molecules associated with the ligand. The decomposition of organic matter starts around  $220\text{ }^{\circ}\text{C}$ . The thermogram of ligand showed first mass loss of 14% (cal. 13.6%) at  $219.3\text{ }^{\circ}\text{C}$  which is due to loss S-atom of ligand molecule. After this TGA curve of ligand molecule showed maximum mass loss of 60% and is almost removed as a volatile components at about  $750\text{ }^{\circ}\text{C}$ . The TGA curves of the complexes showed that the initial mass loss occurring within  $100\text{--}120\text{ }^{\circ}\text{C}$  range is interpreted as loss of moisture and hydrated water molecules during the chelate drying process and the second weight loss at around  $200\text{ }^{\circ}\text{C}$  range is due to loss of coordinated water molecules.

TGA curves of  $[\text{Zn(II)}\text{L}_2(\text{CH}_3\text{COO})_2]\cdot 3\text{H}_2\text{O}$  complex showed first mass loss of 7.9% (cal=7.5%) at  $89.9\text{ }^{\circ}\text{C}$  due to the loss of three hydrated water molecules. The second mass loss of 36% occurring at  $315\text{ }^{\circ}\text{C}$  is due to the decomposition of two (2-methyl-benzimidazole) moieties. The third mass loss of 8.8% is due to the loss of one coordinated acetate ion. The final mass loss of 13% at  $798\text{ }^{\circ}\text{C}$  is due to the conversion Zn metal ion into ZnO.

The  $[\text{SnL}_2\text{Cl}_2]\cdot 2\text{H}_2\text{O}$  complex shows a first weight loss of 4.00% (calcd.4.21%) at  $150\text{--}220\text{ }^{\circ}\text{C}$ , indicating the loss of two hydrated water molecules. The second weight loss of 81.90% (81.98%) between  $220\text{--}500\text{ }^{\circ}\text{C}$  corresponds to the loss of a ligand and two chloride ions. When heated above  $500\text{ }^{\circ}\text{C}$ , a plateau is obtained due to the formation of stable SnO. The residue 16.40 % (calcd. 16.50%) agrees with the analytical result for the metal content.

### Electronic spectra and magnetic properties

The Electronic spectra of the ligand and complexes was recorded in  $10^{-5}\text{ M}$  solution in DMSO. The probable assignments are included in Table 5. The Electronic spectra of Fe(III) complex display two prominent bands<sup>10</sup>. A low intensity broad band around  $12987\text{ cm}^{-1}$  and  $16393\text{ cm}^{-1}$  is assignable to  ${}^2\text{T}_{2\text{g}} \rightarrow {}^2\text{E}_{\text{g}}$  transition<sup>10,15</sup>. Another high intensity band at  $25562\text{ cm}^{-1}$  is due to ligand - metal charge transfer. On the basis of electronic spectra distorted octahedral geometry around Fe(III) ion is suggested. The F(III) complex showed magnetic moment 4.75 BM, is slightly higher than the spin-only value 4.75 BM expected for one unpaired electron, which offers possibility of an octahedral geometry<sup>15</sup>.

**Table 5.** UV-Vis spectra of free ligand and its metal complexes in  $10^{-5}\text{ M}$  solution in DMSO solvent

Compounds	$\lambda_{\text{max}}$ nm	ABS	Wave number $\text{cm}^{-1}$	$\epsilon$ $\text{L.mol}^{-1}.\text{cm}^{-1}$	Transitions
MBIMOT(L)	274	0.663	36496	15809	$\text{n} \rightarrow \pi^*$
	320	0.435	31250	10372	$\text{n} \rightarrow \pi^*$
	262	0.350	38167	22820	(L-M,C.T)
	308	0.288	32467	18777	(L-M,C.T)
Fe(III)-MBIMOT	390	0.035	25641	2282	${}^2\text{T}_{2(\text{D})} \rightarrow {}^2\text{E}_{(\text{D})}$
	460	0.025	21739	1630	${}^5\text{E}_{(\text{D})} \rightarrow {}^5\text{T}_{2(\text{D})}$
	610	0.026	16393	1695	d-d
	770	0.026	12987	1695	d-d
Hg(II)-MBIMOT	276	0.556	36231	39865	(L-M,C.T)
	315	0.410	31746	29397	(L-M,C.T)
	390	0.031	25641	2222	(L-M,C.T)

Sn(II)-MBIMOT	253	0.327	39692	13450	(L-M,C.T)
	275	0.335	36774	13650	(L-M,C.T)
	325	0.275	31255	11075	(L-M,C.T)
	395	0.038	25645	1438	(L-M,C.T)
Zn(II)-MBIMOT	252	0.328	39682	13448	(L-M,C.T)
	272	0.333	36764	13653	(L-M,C.T)
	320	0.270	31250	11070	(L-M,C.T)
	390	0.035	25641	1435	(L-M,C.T)

### Biological activities

The antimicrobial activity of the ligand and the complexes were determined by the disc diffusion technique<sup>16-18</sup>. The compounds were screened in vitro against *Escherichia coli* and *Salmonella paratyphi A*. A 1 mg/mL solution in DMSO was used. The standard used was gentamycin sulphate. Media Used: Peptone-10 g, NaCl-10 g and Yeast extract 5 g, Agar 20 g in 1000 mL of distilled water.

Initially, the stock cultures of bacteria were revived by inoculating in broth media and grown at 37 °C for 18 h<sup>17-19</sup>. The agar plates of the above media were prepared and wells were made in the plate. Each plate was inoculated with 18 h old cultures (100 µL, 10<sup>4</sup> cfu) and spread evenly on the plate. After 20 min, the wells were filled with of compound at different concentrations. The control wells with Gentamycin were also prepared<sup>17-19</sup>. All the plates were incubated at 37 °C for 24 h and the diameter of inhibition zone were noted. The Table 6 contains diameter of inhibition zones (in cm).

**Table 6.** Results of Antibacterial activities of ligand (MBIMOT) and its metal complexes

#### *Escherichia coli*

Compound	100 µg	200 µg	500 µg	MIC µg			
MBIMOT	1.0	1.5	1.8	>500			
Fe (III)MBIMOT	0.3	0.6	0.9	>500			
Hg(II) MBIMOT	0.2	0.4	0.6	>500			
Sn (II)MBIMOT	0.3	0.6	0.8	>500			
Zn (II)MBIMOT	0.2	0.4	0.6	>500			
	25 µg	50 µg	100 µg	200 µg	400 µg	800 µg	MIC µg
Gentamycin	1.8	2.1	2.7	2.9	3.2	3.4	25

#### *Salmonella paratyphi A*

Compound	100 µg	200 µg	500 µg	MIC µg			
MB IMOT	0.6	0.9	1.5	100			
Fe (III)MBIMOT	0.2	0.4	0.6	200			
Hg(II) MBIMOT	0.1	0.2	0.3	>500			
Sn(II)MB IMOT	0.1	0.3	0.6	200			
Zn(II)MBIMOT	0.3	0.6	0.9	200			
	25 µg	50 µg	100 µg	200 µg	400 µg	800 µg	MIC µg
Gentamycin	0.2	1.3	1.6	2.1	2.5	2.7	25

### Conclusion

The ligand molecule acts as a bidentate ligand. The spectroscopic results show the involvement of C=N(at position 3) and C=S(at position 2) groups in coordination to the central metal ion. On the basis of various techniques such as IR, <sup>1</sup>H-NMR, Mass, ESR, UV-Vis

Molar conductance and Thermal studies used for the characterization of metal-complexes a rhombic octahedral geometry for Fe(III) complex and octahedral geometry for Hg(II), Sn(II) and Zn(II) complex is proposed. In view of the foregoing discussions, the high melting points and insolubility in common organic solvents, we have assigned following probable structure of the complexes<sup>10</sup> (Figure 2).

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