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**SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF SCHIFF BASE COMPLEXES OF TRIPOSITIVE AND DIPOSITIVE TRANSITION METAL COMPLEXES.**

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NAGDA G. EL KHOLI

*Chemistry Department, Faculty of Science, Al-Azhar University (Girls), Nasr City, Cairo, Egypt.*

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**Abstract**

A novel tetradentate Schiff base (I) synthesized from sulfametrole [ $N^1$ -(4-methoxy-1,2,5-thiadiazole-3-yl)sulfanilamide] and salicylaldehyde forms stable complexes with transition metal ions such as  $Fe^{(III)}$ ,  $Fe^{(II)}$ ,  $Co^{(II)}$ ,  $Ni^{(II)}$ ,  $Cu^{(II)}$ ,  $Zn^{(II)}$ ,  $Cd^{(II)}$  and  $UO_2^{(II)}$  in absolute ethanol. Elemental analyses, IR,  $^1H$ NMR, electronic spectra, magnetic moment, mass spectra and thermal analysis (TGA) were used to confirm the structures. From the elemental analyses data, the complexes were proposed to have the general formulae  $[M_2Cl_3(I)(H_2O)_5].YH_2O$  (Where  $M=Co^{(II)}, Ni^{(II)}, Cu^{(II)}, Zn^{(II)}$  and  $Cd^{(II)}$ ,  $Y=0-3$ ),  $[Fe_2Cl_5(I)(H_2O)_3].2H_2O$ ,  $(FeSO_4)_2(I)(H_2O)_4.2H_2O$  and  $[(UO_2)_2(NO_3)_3(I)(H_2O)].2H_2O$ . The molar conductance data reveal that all the metal complexes were non-electrolytes. The IR,  $^1H$ NMR, spectra show that ligand (I) is coordinated to the metal ion in a bidentate manner ON and via NO donor sites of the azomethine-N, phenolic-OH, enolic sulfonamide-OH and thiadiazole-N. The magnetic and electronic spectra showed that the complexes exhibit octahedral geometry. The thermal behaviour of the complexes was studied. All the prepared compounds were screened for their antibacterial and antifungal properties and have exhibited potential activities

**Keywords:** Schiff base, transition metal complexes, IR,  $^1H$ NMR, conductance, electronic spectra, magnetic moment, thermal analysis, and biological activity

**Introduction**

Compounds containing N,O and S donor groups and their metal complexes play a key role to understand the coordination chemistry of the transition metal ions<sup>(1,2)</sup>. This is due to remarkable biological activities observed for these compounds, which has since been shown to be related to their metal complexing ability. These compounds present a great variety of biologically active compounds ranging from antitumor, fungicide, bactericide, anti-inflammatory, antiviral activities<sup>(3-5)</sup>. Sulfonamides were the first drugs found to act selectively and could be used systematically as preventive and therapeutic agents against various diseases<sup>(6,7)</sup>.

The condensation products of sulfa drugs with aldehydes, ketones or their derivatives are very active biologically, besides having good complexing ability. Their activity increases on complexation with metal ions<sup>(8)</sup>.

The organotin (IV) complexes of the Schiff bases derived from condensation of 2-acetylfuran, 2-acetyl thiophene, 2-hydroxy-1 naphthaldehyde and benzoylacetone with various sulfa drugs, namely sulfadiazole, sulfadiazine, sulfasoxazole and sulfaguanidine have been prepared<sup>(9)</sup>. The stereo-chemical and biochemical aspects of the complexes of sulfonamide imine of silicon and tin have been also studied<sup>(10)</sup>. Metal complexes of Schiff bases derived from condensation of O-vanillin and 2-thiophene carboxaldehyde and with sulfa drug are reported and characterized<sup>(7,10)</sup>. In the present article Fe<sup>(III)</sup>, Fe<sup>(II)</sup>, Co<sup>(II)</sup>, Ni<sup>(II)</sup>, Cu<sup>(II)</sup>, Zn<sup>(II)</sup>, Cd<sup>(II)</sup> and UO<sub>2</sub><sup>(II)</sup> complexes with a Schiff bases derived from salicylaldehyde and sulphamethoxazole [N'(4-methoxy-1,2,5-thiadiazole-3-yl)] sulfanilamide] are studied.

## Experimental

### Materials

All the chemicals used were of Analar grade, Metals were purchased from E.Merck and were used as received. All solvents used were of standard spectroscopic grade.

### Instrument:

Elemental analyses were carried out at the Microanalytical Research Center (Faculty of Science, Cairo University, Egypt). Metal content were determined complexometrically by standard EDTA titration<sup>(11)</sup>. IR spectra were recorded on Shimadzu FT-IR spectrometer using KBr discs. <sup>1</sup>HNMR spectra were determined on a Varian Gemini 200 MHz spectrometer using DMSO-*d*<sub>6</sub> as solvent and tetramethylsilane (TMS) as an internal reference. The ultraviolet spectra were recorded on a Perkin Elmer Lambda-3BUV-vis spectrometer. The solid reflectance spectra were obtained using a Shimadzu PC<sub>3</sub>101UV-vis-NIR scanning spectrophotometer. The magnetic susceptibilities of the complexes in the solid state were recorded on Sherwood scientific magnetic susceptibility balance. Thermogravimetric analyses (TGA) were recorded on a shimadzu TGA-50H. TGA was carried out in a dynamic nitrogen atmosphere (20 ml min<sup>-1</sup>) at a heating rate of 10°C min<sup>-1</sup>. Mass spectra were obtained by a finnigen SSQ7000 and Shimadzu Ge Ms-QP 100 EX mass spectrometer (Shimadzu, Japan), using the direct inlet system, at the National Research Centre and Cairo University (Egypt).

The molar conductance of the complexes in DMF were measured using sybron-Barnstead conductometer (Meter-PM.6, E=3406). The biological activity (antibacterial and antifungal activities) were carried out at the fermentation biotechnology & applied microbiology (FERM-BAM) Center, Al-Azhar University.

*Synthesis of ligand (I):*

A hot solution of salicylaldehyde (1.22 g ,0.1mol) was mixed with hot solution of sulfametrole (2.86 g, 0.1 mol) in 50ml absolute ethanol. The mixture then left under reflux for 2h. The obtained product was separated by filtration, crystallize from ethanol and washed with diethyl-ether then dried under vacuo. Orange bright crystals were formed, giving 70% yield (Table 1).

*Synthesis of the complexes:*

Metal complexes of Schiff base (I<sub>a-h</sub>) were prepared by addition of hot solution of metal chloride or sulfate or nitrate (0.1 mol) in absolute ethanol to well stirred hot ethanolic solution of the ligand (I) (1.95gm, 0.05 mol). The reaction mixture was stirred under reflux for one hour where upon the complexes precipitated. They were collected by filtration, washed with ethanol and diethyl ether. The analytical data for C,H, N and S are listed in (Table I).

**Table 1. Analytical and physical data of (I) and its complexes.**

Compound	M.p. (°C)	Colour (% yield)	% Found (Calcd.)					$\mu_{\text{eff}}$ (B.M.)	$\Lambda_m$ $\Omega^{-1}\text{mol}^{-1}$ $\text{cm}^2$	
			C	H	N	S	M			
C <sub>16</sub> H <sub>14</sub> N <sub>4</sub> O <sub>4</sub> S <sub>2</sub>	I	>160	Bright orange (87)	49.53 (49.23)	3.42 (3.59)	14.55 (14.36)	16.67 (16.41)	--	--	
[(FeSO <sub>4</sub> ) <sub>2</sub> (I)(H <sub>2</sub> O) <sub>4</sub> ]·2H <sub>2</sub> O C <sub>16</sub> H <sub>26</sub> Fe <sub>2</sub> N <sub>4</sub> O <sub>18</sub> S <sub>4</sub>	I <sub>a</sub>	>300	Dark brown (58)	24.16 (23.94)	3.62 (3.24)	6.49 (6.98)	8.23 (7.98)	13.65 (13.96)	4.73	10.96
FeCl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ]·2H <sub>2</sub> O C <sub>16</sub> H <sub>23</sub> Cl <sub>3</sub> Fe <sub>2</sub> N <sub>4</sub> O <sub>9</sub> S <sub>2</sub>	I <sub>b</sub>	>300	Yellowish brown (55)	25.88 (26.21)	2.92 (2.59)	7.33 (7.65)	8.59 (8.74)	15.46 (15.29)	5.7	12.37
Co <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ]·3 H <sub>2</sub> O C <sub>16</sub> H <sub>29</sub> Cl <sub>3</sub> Co <sub>2</sub> N <sub>4</sub> O <sub>12</sub> S <sub>2</sub>	I <sub>c</sub>	>300	Pale pink (75)	24.96 (25.35)	3.62 (3.83)	7.53 (7.39)	8.68 (8.45)	15.24 (15.58)	4.18	17.11
[Ni <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ]·2H <sub>2</sub> O C <sub>16</sub> H <sub>27</sub> Cl <sub>3</sub> N <sub>4</sub> Ni <sub>2</sub> N <sub>4</sub> O <sub>11</sub> S <sub>2</sub>	I <sub>d</sub>	>300	Yellowish green (69)	25.28 (25.65)	3.40 (3.61)	7.62 (7.48)	8.38 (8.55)	17.31 (16.98)	3.01	9.88
[Cu <sub>2</sub> Cl <sub>3</sub> (H <sub>2</sub> O) <sub>3</sub> ]·2 H <sub>2</sub> O C <sub>16</sub> H <sub>27</sub> Cl <sub>3</sub> Cu <sub>2</sub> N <sub>4</sub> O <sub>11</sub> S <sub>2</sub>	I <sub>e</sub>	>300	Blue (65)	25.72 (25.96)	3.92 (3.65)	7.19 (7.57)	8.29 (8.65)	15.49 (15.96)	1.78	10.45
[Zn <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ] C <sub>16</sub> H <sub>23</sub> Cl <sub>3</sub> N <sub>4</sub> O <sub>9</sub> S <sub>2</sub> Zn <sub>2</sub>	I <sub>f</sub>	>300	White (70)	26.49 (26.83)	2.89 (3.21)	7.63 (7.83)	8.79 (8.94)	17.53 (17.75)	diam.	16.53
[Cd <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ] C <sub>16</sub> H <sub>23</sub> Cd <sub>2</sub> Cl <sub>3</sub> N <sub>4</sub> O <sub>9</sub> S <sub>2</sub>	I <sub>g</sub>	>300	White (73)	24.05 (23.72)	2.90 (2.84)	6.58 (6.92)	7.67 (7.91)	27.35 (27.67)	diam	18.44
[(UO <sub>2</sub> ) <sub>2</sub> (I)(H <sub>2</sub> O)(NO <sub>3</sub> ) <sub>2</sub> ]·2H <sub>2</sub> O C <sub>16</sub> H <sub>19</sub> N <sub>6</sub> O <sub>17</sub> S <sub>2</sub> U <sub>2</sub>	I <sub>h</sub>	>300	Yellowish orange (65)	16.76 (16.42)	1.45 (1.63)	4.62 (4.79)	5.63 (5.47)	---	diam	10.94

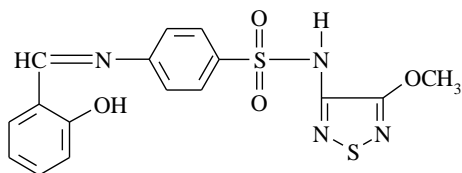
## Results and Discussion

### *The ligand*

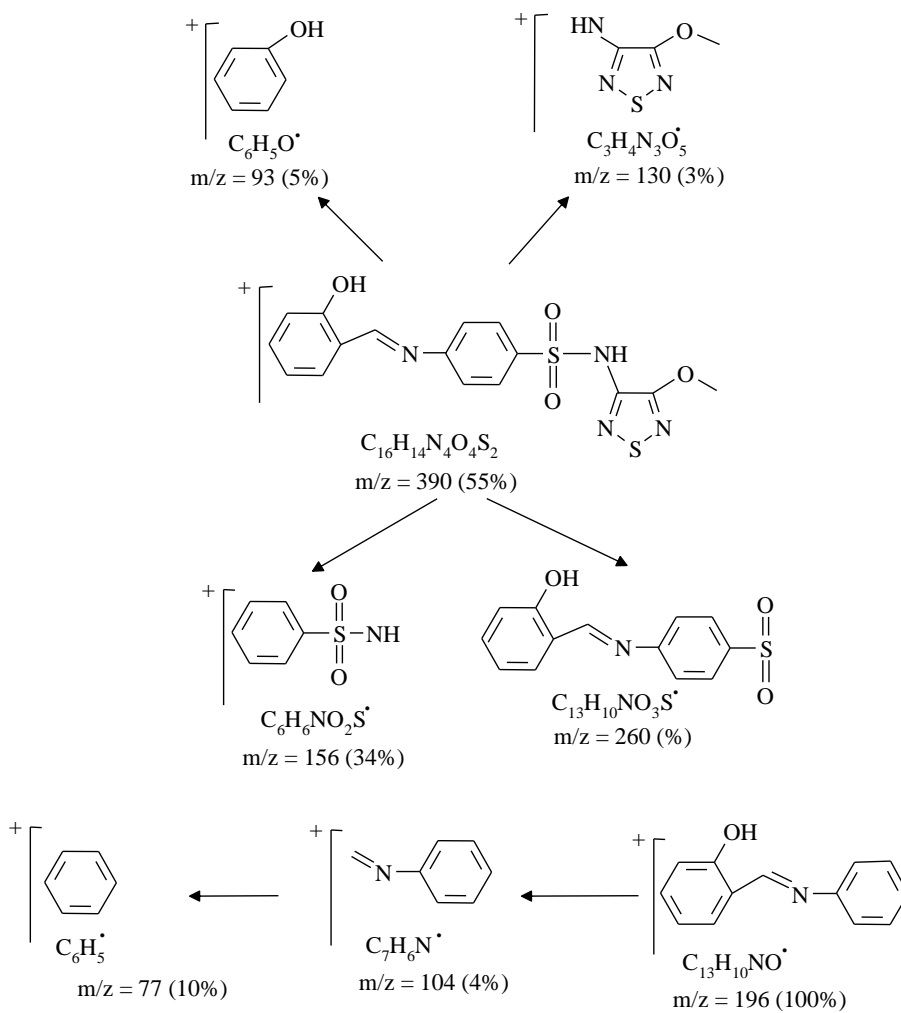
In the present study, we have been synthesized ligand (I) formed from the condensation of the sulfametrole with salicylaldehyde. It is characterized based on elemental analyses C,H,N and S (Table I). The obtained results are in good agreement with those calculated for the suggested formula (Fig. I).

The  $^1\text{H}$ NMR spectrum of the ligand (I) confirms its formulation by the presence of CH=N proton signal at  $\delta = 10.25$  ppm. This was further confirmed by the appearance of stretching vibration band characteristic of  $\nu(\text{CH}=\text{N})$  at  $1620\text{cm}^{-1}$ . Also, the  $^1\text{H}$ NMR spectrum of the ligand exhibits signals at  $\delta = 3.99$ ppm, characteristic for  $\text{OCH}_3$ , aromatic protons at  $\delta=6.56, 7.89$ ppm, and signals at  $\delta=10.6, 12.73$  ppm, characteristic for NH,OH salicylic protons respectively which are exchangable with  $\text{D}_2\text{O}$ .

The structure of this ligand was also confirmed from its mass spectra. The mass spectrometric fragmentation pattern of ligand (I) (Scheme1) showed a base peak at  $m/z = 390$  (55%), together with peaks at  $m/z = 196$  (100%) ,  $m/z = 156$  (34%),  $m/z = 77$  (10%),  $m/z = 260$  (5%),  $m/z=93$  (5%),  $m/z=104$  (4%) and  $m/z=130$  (3%) which confirm the proposed structure of ligand (I) (Fig 1).



**Fig. 1. The suggested structure of ligands (I)**



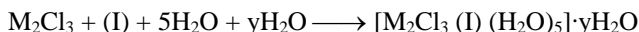
**Scheme 1.** The possible fragmentation pathway of ligand (I)

*The complexes:*

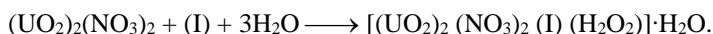
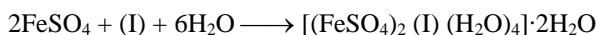
The chemical behaviour of Schiff base (I) towards some transition metal ions namely,  $\text{Fe}^{\text{III}}$ ,  $\text{Fe}^{\text{II}}$ ,  $\text{Ni}^{\text{II}}$ ,  $\text{Cu}^{\text{II}}$ ,  $\text{Zn}^{\text{II}}$ ,  $\text{Cd}^{\text{II}}$  and  $\text{UO}_2^{\text{II}}$  was studied.

Ligand (I) reacts with the metal ions using ethanol as solvent in 1:2 molar ratio. The isolated Schiff base (I) complexes were subjected to elemental analyses (C,H,N,S and metal), IR,  $^1\text{H}$ NMR, UV-vis, magnetic susceptibility, molar conductance and thermal analysis to identify their molecular structure. The

microanalytical data are listed in (Table I), which agree well with the proposed structures according to the following general equation:



Where M : CO<sup>(II)</sup>, Ni<sup>(II)</sup>, Cu<sup>(II)</sup>, Zn<sup>(II)</sup>, Cd<sup>(II)</sup> ; y = 0-3



Further, confirmation of the proposed structures of the adducts of the complex compounds was obtained by the physicochemical studies as shown below.

#### *Molar Conductance:*

The molar conductance measured in DMF of 10<sup>-3</sup>M solutions of these complexes (I<sub>a-h</sub>) fall in the range 9.88-18.44 ohm<sup>-1</sup>cm<sup>2</sup>mol<sup>-1</sup> indicating their nonelectrolytic behaviour<sup>(12)</sup>.

#### *IR spectra:*

The stretching vibration bands of the isolated products (I<sub>a-h</sub>) were assigned using a comparison method of infrared spectra comparing the spectra of the free ligand and its metal complexes (Table 2).

The normal stretching absorption band of the sulfon group [ $\nu_{\text{asym}}(\text{SO}_2)$ ] and [ $\nu_{\text{sym}}(\text{SO}_2)$ ] at 1335cm<sup>-1</sup> and 1159 respectively were shifted to lower frequencies at 1316-1328cm<sup>-1</sup> and 1139-1152cm<sup>-1</sup> respectively. This may be attributed to the transformation of the sulfonamide (-SO<sub>2</sub>NH) to give the enol form (-SO(OH)=N) as a result of complex formation to give more stable six membered ring<sup>(13)</sup>

Also, the stretching vibration band characteristic for  $\nu(\text{NH})$  at 3380cm<sup>-1</sup> in the free ligand, was disappeared on the isolated complexes (I<sub>a-h</sub>). The disappearance of this peak may be attributed to the transformation of the sulfonamide to the enol form as a result of complex formation<sup>(11,15)</sup>. This transformation would result in the partial loss of the amide proton and appearance of the stretching vibration signal for enol (OH) at 3139-3257 cm<sup>-1</sup> for complexes (I<sub>a-h</sub>).



In all metal complexes, there are new bands at lower frequencies between 521-558 and 418-497 cm<sup>-1</sup> which were assigned for  $\nu(\text{M}-\text{O})$  and  $\nu(\text{M}-\text{N})$  stretching mode which are not present in the free ligand (I)<sup>(14)</sup>, (Table 2)

**Table 2. Characteristic IR stretching vibration bands (cm<sup>-1</sup>) of ligand (I) and its metal complexes (I<sub>a</sub> – I<sub>h</sub>)**

Compound	$\nu(\text{NH})$	$\nu(\text{OH})$ enolic	$\nu(\text{CH})$ aromatic	$\nu(\text{CH}=\text{N})$	$\nu(\text{SO}_2)$ (asym.)	$\nu(\text{SO}_2)$ (Sym)	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$	$\nu(\text{M}-\text{Cl})$
I	3380br	--	--	1620 sh	1335s	1159sh	--	--	--
I <sub>a</sub>	--	3253	3044br	1619s	1316w	1152m	552sh	497w	388s
I <sub>b</sub>	--	3139	3041m	1626w	1325s	1129sh	526m	453s	390w
I <sub>c</sub>	--	3146	3053m	1599sh	1327m	1143s	522br	424w	407w
I <sub>d</sub>	--	3200	3059w	1618sh	1327s	1144m	555m	465br	383br
I <sub>e</sub>	--	3154	3055w	1591m	1328br	1157sh	521br	418m	383w
I <sub>f</sub>	--	365	3040sh	1596sh	1317sh	1131br	551w	422br	410s
I <sub>g</sub>	--	3257	3050s	1602br	1323m	1134sh	558s	453m	407br
I <sub>h</sub>	--	3205	3051m	1618br	1325m	1139m	523s	451m	423br

s: strong      m=medium      w= weak      br=braad      sh=sharp

#### <sup>1</sup>HNMR spectra:

The <sup>1</sup>HNMR spectra of the diamagnetic Zn<sup>(II)</sup> and Cd<sup>(II)</sup> complexes showed the same characteristic proton signals for the ligand. The phenolic–OH signal, appeared in the spectrum of ligand (I) at  $\delta$  12.73 ppm is completely disappeared in the spectra of Zn<sup>(II)</sup> and Cd<sup>(II)</sup> complexes indicating that the OH proton is removed by the chelation with Zn<sup>(II)</sup> and Cd<sup>(II)</sup> ions.

Also, the NH proton signal of the free ligand at 10.6 ppm disappeared in complexes and a new signal appeared at  $\delta$  5.4 ppm and 5.5 ppm for Zn<sup>(II)</sup> and Cd<sup>(II)</sup> complexes respectively due to enolization for the ligand on complexes.

The signal observed at  $\delta$  3.86 and 3.32 ppm corresponding to five water molecules in Zn<sup>(II)</sup> and Cd<sup>(II)</sup> complexes, (Table 3).

From the IR and <sup>1</sup>HNMR studies, we suggest that enolization for the ligand occurs in complexation.





*Electronic spectra and magnetic properties:*

The UV-vis spectra of the ligand and its metal complexes are recorded in DMF solution. The spectrum of ligand (I) shows two bands at 308 and 345 nm which due to  $n - \pi^*$  phenolic-OH and azomethine moieties, respectively. These bands are shifted on complexes to lower wavelengths in the region 300-304 nm and 324-326 nm with respect to the ligand, depending on the type of metal ion present in complexes. This indicates the coordination of phenolic-OH and azomethine moieties to metal ions. The spectra of the complexes further display a bands in the ranges of 425-522nm and 575-657 nm, which might be assigned to charge transfer transition from the ligand to metal ions(L  $\rightarrow$  M)<sup>(15)</sup>

The solid reflectance of the Fe<sup>(III)</sup> complex (I<sub>a</sub>) displays band at 21,551 cm<sup>-1</sup>, which may be assigned to the <sup>6</sup>A<sub>1g</sub>  $\rightarrow$  T<sub>2g</sub> (G) transition, also, the <sup>6</sup>A<sub>1g</sub>  $\rightarrow$  <sup>1</sup>T<sub>1g</sub> transition appears as two bands at 17,361 and 15,710 cm<sup>-1</sup> which confirmed the octahedral geometry of the complex<sup>(16)</sup>.

The observed magnetic moment of Fe<sup>(III)</sup> complex is 5.70 B.M. confirmed its octahedral geometry.

The solid reflectance of Fe<sup>(II)</sup> complex (I<sub>b</sub>) showed two bands at 15,673, 17,391 cm<sup>-1</sup> which are assigned to <sup>5</sup>T<sub>2g</sub>(D)–<sup>5</sup>E<sub>g</sub> transition<sup>(17)</sup> and a band at 22,988 cm<sup>-1</sup> is assigned to L  $\rightarrow$  M transfer. The observed magnetic moment of the complex 4.73 B.M.<sup>(18)</sup>, is consistent with octahedral geometry<sup>(17)</sup>. The solid reflectance of the Co<sup>(II)</sup> complex (I<sub>c</sub>) give three bands at 15,330, 17,421 and 21,882 cm<sup>-1</sup> assigned to <sup>4</sup>T<sub>1g</sub> (F)  $\rightarrow$  <sup>4</sup>E<sub>2g</sub> (F), <sup>4</sup>T<sub>1g</sub> (f)  $\rightarrow$  <sup>4</sup>A<sub>2g</sub> (f) and <sup>4</sup>T<sub>1g</sub>  $\rightarrow$  <sup>4</sup>T<sub>2g</sub>(p) transitions respectively, suggesting octahedral geometry around Co(II) ion<sup>(14)</sup>. The magnetic susceptibility lies at 4.18 B.M. which confirms the octahedral structure<sup>(19,20)</sup>.

The solid reflectance of the Ni<sup>(II)</sup> (I<sub>d</sub>) complex shows spectral bands at 14,705, 17,391 and 23,502 cm<sup>-1</sup> assigned to <sup>3</sup>A<sub>2g</sub> $\rightarrow$  <sup>3</sup>T<sub>2g</sub>, <sup>3</sup>A<sub>2g</sub>  $\rightarrow$  <sup>3</sup>T<sub>1g</sub> (f) and <sup>3</sup>A<sub>2g</sub>  $\rightarrow$  <sup>3</sup>T<sub>1g</sub> (p) transitions, respectively. The magnetic moment of 3.01 B.M. fall within the range normally observed for octahedral Ni<sup>(II)</sup> complex<sup>(21,22)</sup>.

The Cu<sup>(II)</sup> complex (I<sub>e</sub>) has a magnetic moment value of 1.78 B.M. which indicates an octahedral geometry. This was supported by the band observed at 15,432 cm<sup>-1</sup> in the solid reflectance spectrum. The band at 22,050 cm<sup>-1</sup> refers to L  $\rightarrow$  M charge transfer<sup>(23)</sup>.

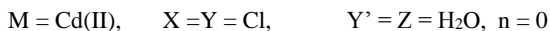
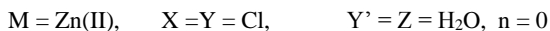
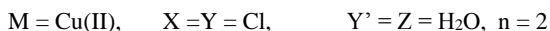
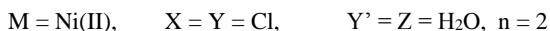
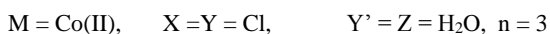
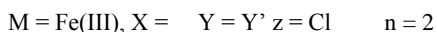
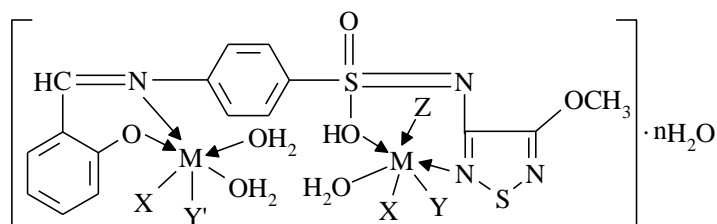
*Thermal Studies*

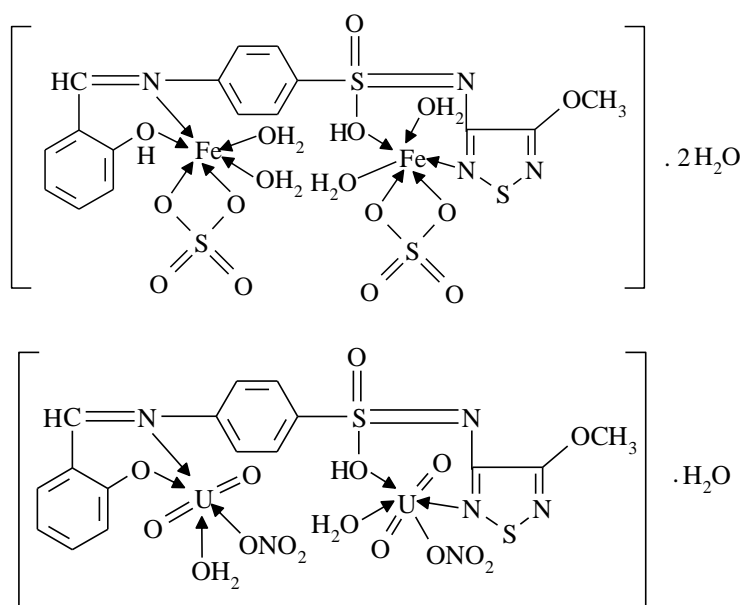
Thermogravimetric studies (TGA) for complex compounds ( $I_{a-h}$ ) were carried out within the temperature range from room temperature up to 1000°C. The determined temperature ranges and percent losses in mass of the solid complexes on heating are given in Table 4. It was observed that lattice and coordinated water were lost relatively at low and high temperatures respectively<sup>(24,25)</sup>. The coordinated water molecules may be eliminated at a separated step as in the case of  $[(FeSO_4)_2(I)(H_2O)_4] \cdot 2H_2O$

However, in the other complexes (Table 4), the coordinated water molecules may be lost accompanied by loss of HCl gas as in  $[Co_2Cl_3(I)(H_2O)_5] \cdot 3H_2O$ ,  $[Ni_2Cl_3(I)(H_2O)_5] \cdot 2H_2O$  and  $[Cu_2Cl_3(I)(H_2O)_5] \cdot 2H_2O$ .

Also in  $[(UO_2)_2(I)(H_2O)(NO_3)_2] \cdot 2H_2O$  the coordinated water molecules are lost accompanied by loss of ligand (I).

From the above thermogravimetric analysis, the overall weight losses for complex compounds ( $I_{a-h}$ ) agree well with the proposed formulae obtained by elemental analyses, IR, <sup>1</sup>HNMR, solid reflectance, magnetic susceptibility and electronic spectra. From the above findings, we suggest the structure of the complexes ( $I_{a-h}$ ) as follow:



Suggested structure of formula ligand (I) complex compounds (I<sub>a-h</sub>)*Biological activity*

The antibacterial and antifungal tests were carried out for the prepared compounds using disc diffuse method(26) at different concentrations 1, 2.5 and 5mg/ml. Chloramphenicol and Grisolvine were used as reference compounds for antibacterial and antifungal activities, respectively. The data are listed in table 5.

It can be seen from table 5 that the antibacterial and antifungal of ligand (I) are similar to that of the standard at certain concentrations. Also some of the complexes showed more or the same antibacterial activity and moderate antifungal activity compared with the ligand (I) and the standards.



**Table 5. Antimicrobial activity of ligand (I) and its complexes**

Test organisms Sample	Conc.		<i>E. coli</i>	<i>Salmonella typhi</i>	<i>Staphylococcus aureus</i>	<i>Bacillus subtilis</i>	<i>Aspergillus terreus</i>	<i>Aspergillus flavus</i>
<b>I</b>	1	(mg/m)	+	++	++	++	+	+
	2.5		++	+++	++	++	++	+
	5		++	+++	++	++	++	++
<b>I<sub>a</sub></b>	1	(m/g/m)	+	+	+	+	+	+
	2.5		+	+	+	+	+	+
	5		+	++	+	+	+	++
<b>I<sub>b</sub></b>	1	(m/g/m)	+	0	+	+	+	+
	2.5		+	+	+	+	+	+
	5		+	+	+	+	+	+
<b>I<sub>c</sub></b>	1	(m/g/m)	+	+	+	+	+	++
	2.5		+	+	++	++	+	+
	5		++	++	++	++	++	+
<b>I<sub>d</sub></b>	1	(m/g/m)	+	+	+++	+	++	++
	2.5		++	++	+	+	+	+
	5		++	++	+	+	+	+
<b>I<sub>e</sub></b>	1	(mg/m)	+	+	+	+	+	+
	2.5		+	++	+	++	+	+
	5		++	++	++	++	+	+
<b>I<sub>f</sub></b>	1	(m/g/m)	+	+	++	+	+	++
	2.5		+	++	+	+	++	+
	5		++	++	+	+	+	+
<b>I<sub>g</sub></b>	1	(mg/m)	+	++	+	+	+	+
	2.5		+	++	++	++	+	+
	5		++	+++	++	++	++	++
<b>ih</b>	1	(m/g/m)	0	+	0	+	+	+
	2.5		+	+	+	+	+	+
	5		+	++	+	+	+	+
<b>St.</b>	1	(mg/m)	++	+++	++	++	+++	+++
	2.5		+++	+++	++	+++	+++	+++
	5		+++	+++	+++	+++	+++	+++

St. = Reference standard; Chloramphenicol was used as a standard antibacterial agent and grisoflavin was used as a standard antifungal

- \* The test was done using the diffusion agar technique
- \* Well diameter 1 cm (100 µl of each conc. Was tested)
- \* Inhibition values = 0.1-0.5 cm beyond control = +
- \* Inhibition values = 0.6 cm beyond control = ++
- \* Inhibition values = 1.1-1.5 cm beyond control = +++
- \* Inhibition values > 1.5 cm beyond control = ++++
- \* 0 = Not detected

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**Table 3. <sup>1</sup>H-NMR data of ligand (I) and Zn<sup>2+</sup>, Cd<sup>2+</sup> metal complexes in ppm.**

Compound	OCH <sub>3</sub>	Aromatic protons	NH proton	CH=N azomethin proton	OH enol proton	OH salicylic	Coordinated water
I	3.99 (s, 3H)	6.56 (d, J = 9 Hz, 2H, Ph-H) 7.89 (d, J=9Hz, 2H, Ph-H)	10.6 (brs, 1H, NH, exchangeable with D <sub>2</sub> O)	10.25 (s, 1H, CH=N)	--	12.73 (s, 1H, OH exchangeable with D <sub>2</sub> O)	--
[Zn <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>5</sub> ]	4.02 (s, 3H)	6.57 (d, J=10.2 Hz, 2H, Ph-H) 7.46 (d, J=10.2 Hz, 2 H, Ph-H)		8.43 (s, 1H, CH=N)	5.4 (brs, 1H, enolic OH, exchagable with D <sub>2</sub> O)		3.86 s, coordinate (H <sub>2</sub> O)
[Cd <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>5</sub> ]	3.94 (s, 3H)	6.46 (d, J = 8.4Hz, 2H, Ph-H) 7.54 (d, J=8.4Hz, 2H, Ph-H)	--	8.92 (s, 1H, CH=N)	5.51 (brs, 1H, enolic OH exchangeable with D <sub>2</sub> O)		3.32 (s, coordinate H <sub>2</sub> O)









**Table 4. Thermogravimetric data of ligand (I) and metal complexes**

Complex	TG range, (°C)	DTG <sub>max</sub> (°C)	Mass loss % Found (calcd)	Total mass loss % Found (calcd)	Assignment	Metallic residue
[Fe <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>3</sub> ·2 H <sub>2</sub> O	30-100 100-270 270-700	70 220 340, 470	4.51 (4.68) 30.49 (30.12) 44.25 (44.37)	79.25 (79.17)	- loss of H <sub>2</sub> O - Loss of 5HCl, /2H <sub>2</sub> and 1.5O <sub>2</sub> - Loss of (I).	Fe <sub>2</sub> O <sub>3</sub>
[FeSO <sub>4</sub> ] <sub>2</sub> (I)(H <sub>2</sub> O) <sub>4</sub> ·2H <sub>2</sub> O	30-280 280-800	120, 250 370, 650	13.22 (13.47) 48.50 (48.63)	61.72 (62.10)	- Loss of 6 H <sub>2</sub> O - Loss of (I)	2 FeSO <sub>4</sub>
[Co <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>5</sub> ]·3H <sub>2</sub> O	30-100 130-380 380-850	90 280 530, 670	7.77 (7.13) 25.76 (25.94) 45.49 (45.02)	79.02 (78.09)	- Loss of 3 H <sub>2</sub> O - Loss of 3 H <sub>2</sub> O, 1/2H <sub>2</sub> , O <sub>2</sub> and 3HCl - Loss of (I)	Co <sub>2</sub> O <sub>3</sub>
[Ni <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>5</sub> ]·2H <sub>2</sub> O	40-150 150-320 320-700	60 290 370, 500	4.78 (4.87) 26.69 (26.57) 43.67 (43.95)	75.14 (75.39)	- Loss of 2 H <sub>2</sub> O. - Loss of 3 H <sub>2</sub> O, 1/2H <sub>2</sub> , O <sub>2</sub> and 3HCl. - Loss of (I)	Ni <sub>2</sub> O <sub>3</sub>
[Cu <sub>2</sub> Cl <sub>3</sub> (I)(H <sub>2</sub> O) <sub>5</sub> ]·2H <sub>2</sub> O	30-120 120-250 250-700	75 210 350, 540	4.84 (4.81) 26.80 (26.25) 47.65 (47.70)	79.29 (78.76)	- Loss of 2 H <sub>2</sub> O. - Loss of 3 HCl, 3H <sub>2</sub> O, 1/2H <sub>2</sub> and O <sub>2</sub> . - Loss of (I)	Cu <sub>2</sub> O
[(UO <sub>2</sub> ) <sub>2</sub> (I)(H <sub>2</sub> O) <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> ·H <sub>2</sub> O	30-130 130-900	70 140, 230, 380, 680	10.91 (10.69) 21.08 (20.36)	31.99 (31.05)	- Loss of 2 H <sub>2</sub> O. - Loss of H <sub>2</sub> O and (I)	2 UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub>









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