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

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Abstract

A novel tetradentate Schiff base (I) synthesized from sulfametrole [N^1-(4-methoxy-1,2,5-thiadiazole-3-y1)sulfanilamide] and salciyldelyde 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, ^{1}HNMR, 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, ^{1}HNMR, 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, ^{1}HNMR, 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(1,2). This is due to remarkable biological activities observed for there 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(3-5). Sulfonamides were the first drugs found to act selectivity and could be used systematically as preventive and therapeutic agents against various diseases(6,7).

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

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 sulfadia-zole, sulfadi-azine, sulfasoxazole and sulfaguanidine have been prepared(9). The stereo-chemical and biochemical aspects of the complexes of sulfonamide imine of silicon and tin have been also studied(10). Metal complexes of Schiff bases derived from condensation of O-vanilin and 2-thiophene carboxaldehyde and with sulfa drug are reported and characterized(7,10). In the present article $Fe^{(III)}$, $Fe^{(II)}$, $Co^{(II)}$, $Ni^{(II)}$, $Cu^{(II)}$, $Zn^{(II)}$, $Cd^{(II)}$ and $UO_2^{(II)}$ complexes with a Schiff bases derived from salicylaldehyde and sulphametrole [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.Merek and were used as received. All solvents used were of standard spectroscopic grade.

Instrument:

Elemental analyses were carried out at the Microanlaytical Research Center (Faculty of Science, Cairo University, Egypt). Metal content were determined complexometrically by standard EDTA titration(11). IR spectra were recorded on Shimadzu FT-IR spectrometer using KBr discs. ^{1}HNMR spectra were determined on a Varian Gemini 200 MHz spectrometer using DMSO-d_6 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_3$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^{-1}) at a heating rate of 10^oC min^{-1}. 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 conductomter (Meter-PM.6, E=3406). The biolog-ical 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 salicyladehyde (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 vaccuo. Orange bright crystals were formed, giving 70% yield (Table 1).

Synthesis of the complexes:

Metal complexes of Schiff base (I_{a-h}) 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 filteration, 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.	Colour	% Found (Calcd.)					μ_{eff}	Λ_m $\Omega^{-1}mol^{-1}$ cm^2
		(°C)	(% yield)	C	H	N	S	M	(B.M.)	
$C_{16}H_{14}N_4O_4S_2$	I	>160	Bright orange (87)	49.53 (49.23)	3.42 (3.59)	14.55 (14.36)	16.67 (16.41)	--	--	--
$[(FeSO_4)_2(I)(H_2O)_4]\cdot 2H_2O$ $C_{16}H_{26}Fe_2N_4O_{18}S_4$	I_a	>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
$FeCl_5(I)(H_2O)_3]\cdot 2H_2O$ $C_{16}H_{23}Cl_5Fe_2N_4O_9S_2$	I_b	>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
$Co_2Cl_3\ (I)(H_2O)_5]\cdot 3\ H_2O$ $C_{16}H_{29}Cl_3Co_2N_4O_{12}S_2$	I_c	>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
$[Ni_2Cl_3(I)(H_2O)_5]\cdot 2H_2O$ $C_{16}H_{27}Cl_3N_4Ni_2N_4O_{11}S_2$	I_d	>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
$[Cu_2Cl_3(H_2O)_5]\cdot 2\ H_2O$ $C_{16}H_{27}Cl_3Cu_2N_4O_{11}S_2$	I_e	>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
$[(Zn_2Cl_3(I)(H_2O)_5]$ $C_{16}H_{23}Cl_3N_4O_9S_2Zn_2$	I_f	>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
$[Cd_2Cl_3(I)(H_2O)_5]$ $C_{16}H_{23}Cd_2Cl_3N_4O_9S_2$	I_g	>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
$[(UO_2)_2(I)(H_2O)(NO_3)_2]\cdot 2H_2O$ $C_{16}H_{19}N_6O_{17}S_2U_2$	I_h	>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}HNMR 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 ν(CH=N) at 1620cm^{-1}. Also, the ^{1}HNMR spectrum of the ligand exhibits signals at $\delta = 3.99$ppm, characteristic for OCH_3, aromatic protons at δ=6.56, 7.89ppm, and signals at δ=10.6,12.73 ppm, characteristic for NH,OH salicylic protons respectively which are exhangable with D_2O.

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).

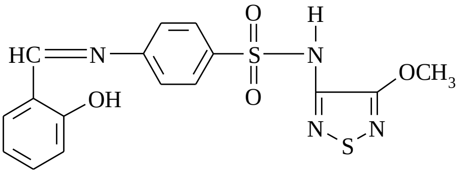
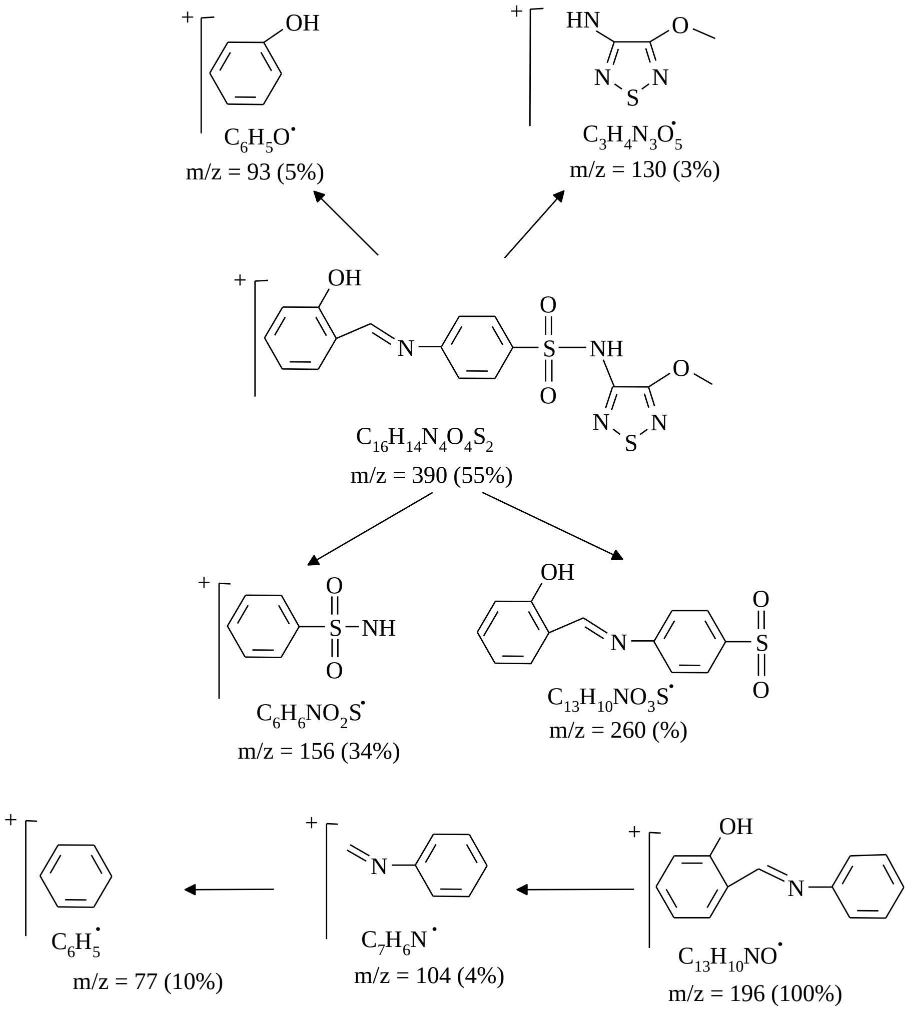


Fig. 1. The suggested structure of ligands (I)



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

The complexes:

The chemical behaviour of Schiff base (I) towards some transition metal ions namely, $Fe^{(III)}$, $Fe^{(II)}$, $Ni^{(II)}$, $Cu^{(II)}$, $Zn^{(II)}$, $Cd^{(II)}$ and $UO_2^{(II)}$ 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}HNMR,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:

$M_2Cl_3 + (I) + 5H_2O + yH_2O \longrightarrow [M_2Cl_3\ (I)\ (H_2O)_5]{\cdot}yH_2O$
Where M : $CO^{(II)}$, $Ni^{(II)}$, $Cu^{(II)}$, $Zn^{(II)}$, $Cd^{(II)}$; y = 0-3
$2FeCl_3 + (I) + 5H_2O \longrightarrow [Fe_2Cl_5(I)\ (H_2O)_3]{\cdot}2H_2O + HCl$
$2FeSO_4 + (I) + 6H_2O \longrightarrow [(FeSO_4)_2\ (I)\ (H_2O)_4]{\cdot}2H_2O$
$(UO_2)_2(NO_3)_2 + (I) + 3H_2O \longrightarrow [(UO_2)_2\ (NO_3)_2\ (I)\ (H_2O_2)]{\cdot}H_2O$.

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

Molar Conductance:

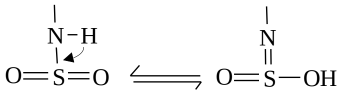
The molar conductance measured in DMF of 10^{-3}M solutions of these complexes ($I_{a–h}$) fall in the range 9.88-18.44 $ohm^{-1}cm^{2}mol^{-1}$ indicating their nonelectrolytic behaviour(12).

IR spectra:

The stretching vibration bands of the isolated products (I_{a-h}) 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 [$\upsilon_{asym}(SO_2)$] and [$\upsilon_{sym}(SO_2)$] at 1335cm^{-1} and 1159 respectively were shifted to lower frequencies at 1316-1328cm^{-1} and 1139-1152cm^{-1} respectively. This may be attributed to the transformation of the sulfonamide ($–SO_2NH$) to give the enol form ($–SO(OH)=N$) as a result of complex formation to give more stable six membered ring(13)

Also, the stretching vibration band characteristic for ν(NH) at 3380cm^{-1} in the free lignad, was disappeared on the isolated complexes (I_{a-h}). The disappearance of this peak may be attributed to the transformation of the sulfonamide to the enol form as a result of complex formation(11,15). 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^{-1} for complexes (I_{a-h}).



In all metal complexes, there are new bands at lower frequencies between 521-558 and 418-497 cm^{-1} which were assigned for ν(M-O) and ν(M-N) stretching mode which are not present in the free lignad (I)(14),(Table 2)

Table 2. Characteristic IR stretching vibration bands (cm^{-1}) of ligand (I) and its metal complexes (I_a – I_h)

Compound	υ(NH)	υ(OH) enolic	υ(CH) aromatic	υ(CH=N)	υ(SO_2) (asym.)	υ(SO_2) (Sym)	υ(M–O)	υ(M–N)	υ(M-Cl)
I	3380br	--	--	1620 sh	1335s	1159sh	--	--	--
I_a	--	3253	3044br	1619s	1316w	1152m	552sh	497w	388s
I_b	--	3139	3041m	1626w	1325s	1129sh	526m	453s	390w
I_c	--	3146	3053m	1599sh	1327m	1143s	522br	424w	407w
I_d	--	3200	3059w	1618sh	1327s	1144m	555m	465br	383br
I_e	--	3154	3055w	1591m	1328br	1157sh	521br	418m	383w
I_f	--	365	3040sh	1596sh	1317sh	1131br	551w	422br	410s
I_g	--	3257	3050s	1602br	1323m	1134sh	558s	453m	407br
I_h	--	3205	3051m	1618br	1325m	1139m	523s	451m	423br

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

^{1}HNMR spectra:

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

Also, the NH proton signal of the free lignas at 10.6 ppm disappeared in complexes and a new signal appeared at δ 5.4 ppm and 5.5 ppm for Zn$^{(II)}$ and Cd$^{(II)}$ complexes respectively due to enolization for the ligand on complexes.

The signal observed at δ 3.86 and 3.32 ppm corresponding to five water molecules in Zn$^{(II)}$ and Cd$^{(II)}$ complexes, (Table 3).

From the IR and ^{1}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 and n - π^* 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)(15)

The solid reflectance of the Fe$^{(III)}$ complex (I$_a$) displays band at 21,551 cm^{-1}, which may be assigned to the $^6A_1g \longrightarrow T_2g$ (G) transition , also, the $^6A_1g \rightarrow {}^1T_1g$ transition appears as two bands at 17,361 and 15,710 cm^{-1} which confirmed the octahedral geometry of the complex(16).

The observed magnetic moment of Fe$^{(III)}$ complex is 5.70 B.M. confirmed its octahedral geometry.

The solid reflectance of Fe$^{(II)}$ complex (I$_b$) showed two bands at 15,673 , 17,391 cm^{-1} which are assigned to $^5T_{2g}$(D)–^5E_g transition(17) and a band at 22,988 cm^{-1} is assigned to L $\rightarrow$ M transfer. The observed magnetic moment of the complex 4.73 B.M(18), is consistent with octahedral geometry(17). The solid reflectance of the Co$^{(II)}$ complex (I$_c$) give three bands at 15,330,17,421 and 21,882 cm^{-1} assigned to ^4T_1g (F) $\rightarrow$ ^4E_2g (F), ^4T_1g (f) $\rightarrow$ ^4A_2g (f) and $^4T_1g \rightarrow {}^4T_2g$(p) transitions respectively, suggesting octahedral geometry around Co(II) ion(14). The magnetic susceptibility lies at 4.18 B.M. which confirms the octahedral structure(19,20).

The solid reflectance of the Ni$^{(II)}$ (I$_d$) complex shows spectral bands at 14,705, 17,391 and 23,502 cm^{-1} assigned to $^3A_2g \rightarrow {}^3T_2g$, $^3A_2g \rightarrow {}^3T_1g$ (f) and $^3A_2g \rightarrow {}^3T_1g$ (p) transitions, respectively. The magnetic moment of 3.01 B.M. fall within the range normally observed for octahedral Ni$^{(II)}$ complex(21,22).

The Cu$^{(II)}$ complex (I$_e$) 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^{-1} in the solid reflectance spectrum. The band at 22,050 cm^{-1} refers to L $\rightarrow$ M charge transfer(23).

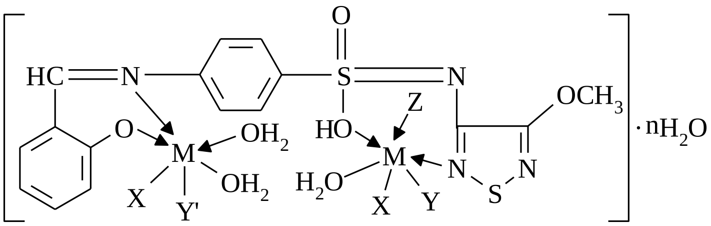
Thermal Studies

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$^{(24,25)}$. 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 $[Co_2Cl_3(I)(H_2O)_5].3H_2O$,$[Ni_2Cl_3(I)(H_2O)_5].2H_2O$ and $[Cu_2Cl_3(I)(H_2O)_5].2H_2O$.

Also in $[(UO_2)_2(I)(H_2O)(NO_3)_2.2H_2O$ 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, ^{1}HNMR, solid reflectance, magnetic susceptibility and electronic spectra. From the above findings, we suggest the structure of the complexes (I_{a-h}) as follow:



M = Fe(III), X =   Y = Y' z = Cl   n = 2

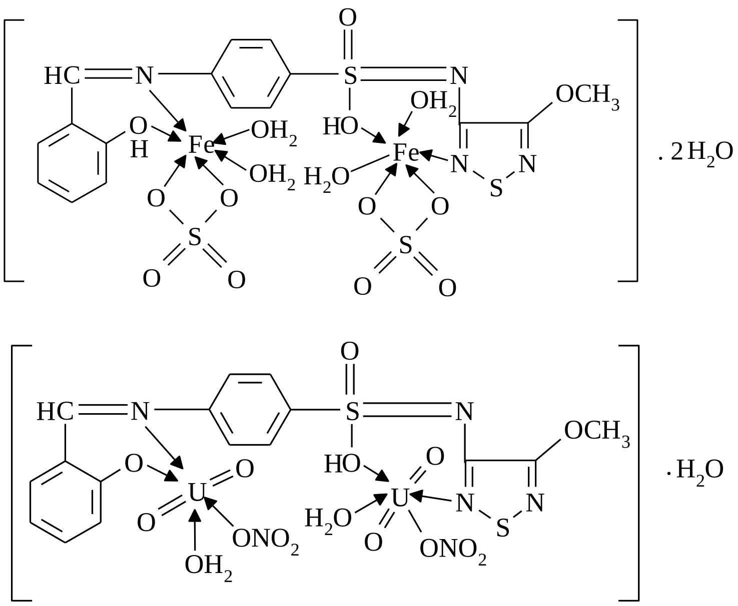
M = Co(II),   X =Y = Cl,   Y' = Z = H_2O, n = 3

M = Ni(II),   X = Y = Cl,   Y' = Z = H_2O, n = 2

M = Cu(II),   X =Y = Cl,   Y' = Z = H_2O, n = 2

M = Zn(II),   X =Y = Cl,   Y' = Z = H_2O, n = 0

M = Cd(II),   X =Y = Cl,   Y' = Z = H_2O, n = 0



Suggested structure of formula ligand (I) complex compounds (I_{a-h})

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 Grisoluvine 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.		*E.coli*	*Salmonella typhi*	*Staphylococcus aureus*	*Bacillus subtillus*	*Aspergillus terrus*	*Aspergillus flafus*
I	1	(mg/m)	+	++	++	++	+	+
	2.5		++	+++	++	++	++	+
	5		++	+++	++	++	++	++
$\mathbf{I_a}$	1	(mg/m)	+	+	+	+	+	+
	2.5		+	+	+	+	+	+
	5		+	++	+	+	+	++
$\mathbf{I_b}$	1	(mg/m)	+	0	+	+	+	+
	2.5		+	+	+	+	+	+
	5		+	+	+	+	+	+
$\mathbf{I_c}$	1	(mg/m)	+	+	+	+	+	++
	2.5		+	+	++	++	+	+
	5		++	++	++	++	++	+
$\mathbf{I_d}$	1	(mg/m)	+	+	+++	+	++	++
	2.5		++	++	+	+	+	+
	5		++	++	+	+	+	+
$\mathbf{I_e}$	1	(mg/m)	+	+	+	+	+	+
	2.5		+	++	+	++	+	+
	5		++	++	++	++	+	+
$\mathbf{I_f}$	1	(mg/m)	+	+	++	+	+	++
	2.5		+	++	+	+	++	+
	5		++	++	+	+	+	+
$\mathbf{I_g}$	1	(mg/m)	+	++	+	+	+	+
	2.5		+	++	++	++	+	+
	5		++	+++	++	++	++	++
Ih	1	(mg/m)	0	+	0	+	+	+
	2.5		+	+	+	+	+	+
	5		+	++	+	+	+	+
St.	1	(mg/m)	++	+++	++	++	+++	+++
	2.5		+++	+++	++	+++	+++	+++
	5		+++	+++	+++	+++	+++	+++

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

* The test was done using the diffusion agar technique

* Well diameter 1 cm (100 µl of eacn 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. ^{1}H-NMR data of ligand (I) and Zn^{2+}, Cd^{2+} metal complexes in ppm.

Compound	OCH_3	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=9H_z, 2H, Ph–H)	10.6 (brs, 1H, NH, exchangeable with D_2O)	10.25 (s, 1H, CH=N)	--	12.73 (s, 1H,OH exchangeable with D_2O	--
$[Zn_2Cl_3(I)(H_2O)_5]$	4.02 (s, 3H)	6.57 (d, J=10.2 H_2, 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_2O)		3.86 s, coordinate (H_2O)
$[Cd_2Cl_3(I)(H_2O)_5]$	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_2O)		3.32 (s, coordinate H_2O)

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

Complex	TG range, (°C)	DTG_{max} (°C)	Mass loss % Found (calld)	Total mass loss % Found (calld)	Assignment	Metallic residue
$[Fe_2Cl_5(I)(H_2O)_3\cdot 2\ H_2O$	30-100	70	4.51 (4.68)	79.25 (79.17)	- loss of H_2O	Fe_2O_3
	100-270	220	30.49 (30.12)		- Loss of 5HCl, /$2H_2$ and $1.5O_2$	
	270-700	340, 470	44.25 (44.37)		- Loss of (I).	
$[FeSO_4)_2(I)(H_2O)_4\cdot 2H_2O$	30-280	120, 250	13.22 (13.47)	61.72 (62.10)	- Loss of 6 H_2O	2 $FeSO_4$
	280-800	370, 650	48.50 (48.63)		- Loss of (I)	
$[Co_2Cl_3(I)(H_2O)_5]\cdot 3H_2O$	30-100	90	7.77 (7.13)		- Loss of 3 H_2O	Co_2O_3
	130-380	280	25.76 (25.94)		- Loss of 3 H_2O, $1/2H_2$, O_2 and 3HCl	
	380-850	530, 670	45.49 (45.02)	79.02 (78.09)	- Loss of (I)	
$[Ni_2Cl_3(I)(H_2O)_5]\cdot 2H_2O$	40-150	60	4.78 (4.87)		- Loss of 2 H_2O.	Ni_2O_3
	150-320	290	26.69 (26.57)		- Loss of 3 H_2O, $1/2H_2$, O_2 and 3HCl.	
	320-700	370, 500	43.67 (43.95)	75.14 (75.39)	- Loss of (I)	
$[Cu_2Cl_3\ (I)(H_2O)_5]\cdot 2H_2O$	30-120	75	4.84 (4.81)		- Loss of 2 H_2O.	Cu_2O
	120-250	210	26.80 (26.25)		- Loss of 3 HCl, $3H_2O$, $1/2H_2$ and O_2.	
	250-700	350, 540	47.65 (47.70)	79.29 (78.76)	- Loss of (I)	
$[(UO_2)_2(I)(H_2O)_2(NO_3)_2\cdot H_2O$	30-130	70	10.91 (10.69)		- Loss of 2 H_2O.	2 $UO_2(NO_3)_2$
	130-900	140, 230,	21.08 (20.36)		- Loss of H_2O and (I)	
		380, 680		31.99 (31.05)		

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