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RESEARCH ARTICLE

Synthesis, Spectral and Antimicrobial Studies of Some d^{10} Metal-Ions Ditellura Tetraazamacrocyclic Complexes

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Abstract: A novel series of some 10 membered ditellura tetraazamacrocyclic complexes of the type $[ML^1Cl_2]$, $[ML^2Cl_2]$ and $[ML^3Cl_2]$ where [L^1, L^2, L^3= 10-membered ditellura tetraazamacrocyclic ligands; M = Zn(II), Cd(II) and Hg(II)] have been synthesized *via* template condensation reactions of 1,2-diaminopropane with diaryltelluriumdichlorides, R_2TeCl_2, (R = *p*- methoxyphenyl, 3-methyl-4-hydroxyphenyl and *p*-hydroxyphenyl) and divalent metal chlorides in dry methanol. The formation of proposed macrocyclic skeletons and their donor sites have been predicted on the basis of elemental analyses, molar conductance and magnetic susceptibility measurements, IR, ^{1}H NMR and electronic absorption spectral studies. On the basis of these studies, a distorted octahedral structure has been suggested for these complexes. These new complexes have also been evaluated for their antimicrobial activity against gram negative and gram positive bacteria and fungi.

Keywords: Ditellura tetraazamacrocycles, Diaryltellurium dichlorides, Template condensation, 1, 2-Diaminopropane,

Introduction

The chemistry of macrocyclic ligands and their metal complexes has become a growing class of research and received the attention of both bioinorganic and inorganic chemists in recent years[1,2]. Due to the presence of various donor atoms in macrocycle and their pliancy and ability to coordinate with metal ions, transition metal complexes are of great importance in synthesis[3]. Synthesis of polyazamacrocyclic complexes containing both 'hard' and 'soft' metal ions and multiple metallocene redox active groups have been reported by Beer *et al*[4]. The macrocyclic complexes are also significant in coordination chemistry due to their biological activity[5-7], applications in biometallic activation and catalysis[8], as models for biochemically important proteins and enzymes[9-13] and as single source precursors in metal-organic chemical vapor deposition processes[14-16]. Coordination behavior of organometallic compounds containing bidentate ligands has been the subject of substantial studies in recent years[17-19].

Synthetic route for the metal complexes with tellurium containing macrocycles have been reported by Srivastava *et al*[20]. Recently template synthesis for transition metal complexes of tellurium containing tetraazamacrocycles[21-23] has been reported. In view of this, we herein report the synthesis, characterization and antimicrobial activity of a new series of Zn(II), Cd(II) and Hg(II) complexes with three 10-membered ditellura tetraazamacrocycles.

Experimental

The chemicals used for the synthesis, recrystallization and analyses were of reagent grade. The solvents, phenol, anisole and *o*-cresol were purified by standard methods[24,25] before use.

The carbon, hydrogen and nitrogen analyses for these metal complexes were obtained from SAIF, Punjab University, Chandigarh on a microprocessor based Thermo Scientific (FLASH 2000) CHN Elemental Analyser. Chlorine and tellurium contents were determined volumetrically[26]. The conductance measurements were performed in acetonitrile at 25±2 °C using a dip type conductivity cell with smooth platinum electrodes (cell constant = 0.997) on a microprocessor based conductivity bridge type MICROSIL.

KBr pellet method was used to obtain Mid-IR (4000-400 cm^{-1}) and H. D. P. E. for far IR(40-500 cm^{-1}) spectra on FT-Infra Red spectrometer model nicolet IS50 (Thermo Scientific). Proton NMR spectra were recorded at SAIF, Panjab University, Chandigarh in DMSO-d$_6$ using TMS as an internal reference on BRUKER AVANCE II 400 NMR spectrometer. Atomic absorption spectrophotometer (ECIL Model No.4129) was used to estimate the metal ions. Electronic spectra were recorded in $BaSO_4$ at 25±2 °C on a UV-Vis-NIR spectrophotometer, model UV-3600 Plus (SHIMADZU). Magnetic mass susceptibility data were obtained from National Physical Laboratory, New Delhi on a Gouy's balance (model Johnson Matthey Alfa products) using glycerin as a calibrant. Melting points were determined in open capillary tube and are uncorrected.

The complexes were screened for *in vitro* antimicrobial activity against gram +ve bacteria: *Staphylococcus aureus* (MTCC 2901), *Bacillus subtilis* (MTCC 2063) and *Bacillus cereus* (MTCC 7350), gram -ve bacteria: *Escherichia coli* (MTCC 1652) and *Salmonella typhi* (ATCC 15499); fungal strains: *Candida albicans* (MTCC 227), *Aspergillus niger* (MTCC 8189) and *Aspergillus fumigatus* (ITCC 4517) using tube dilution method[27]. The test and standard compound were both serially diluted in double strength nutrient broth- I.P for bacteria and Sabouraud Dextrose Broth –I.P for fungi[28]. Dilutions were made in DMSO. Cefadroxil (antibacterial agent) and fluconazole (antifungal agent) were used as control. The cultures were incubated for a time period of 24 h at 37±2 °C for bacteria and for fungal strains at 25±1 °C for 7 days (*A. niger*), 37 ±2 °C for 48 h (*C. albicans*), 35±2 °C for 72 h (*A. fumigatus*) and the results were stated in terms of minimum inhibitory concentration (MIC). MIC is the lowest concentration of an antimicrobial agent that prevents the viable growth after required incubation.

Preparation of diaryltelluriumdichlorides

Bis(*p*-hydroxyphenyl)tellurium(IV)dichloride[29], bis(3-methyl-4-hydroxyphenyl) tellurium(IV) dichloride[30] and bis(*p*-methoxyphenyl) tellurium(IV) dichloride[31,32] were prepared by direct reactions of $TeCl_4$ with phenol, *o*-cresol and anisole respectively, as reported in the literature.

Synthesis of metal complexes with 10-membered tellurium tetraazamacrocycles (Te_2N_4M system)

The metal complexes were prepared by the template condensation reaction of diaryltelluriumdichlorides and 1,2-diaminopropane with metal chlorides in 2: 2: 1 molar ratios.

General method for metal complexes

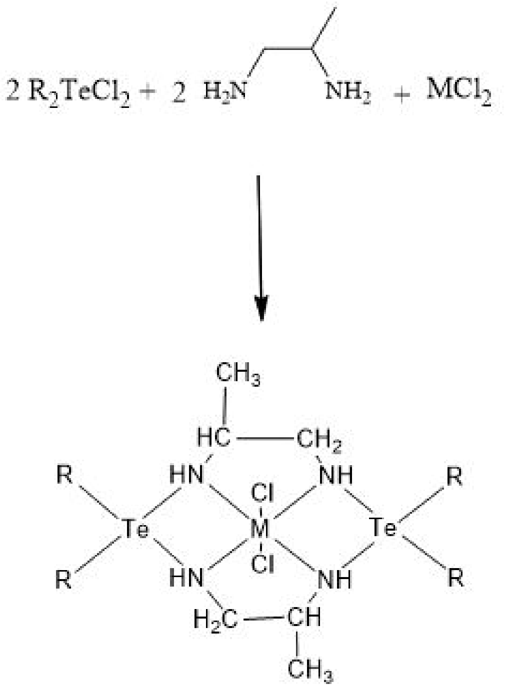
A saturated hot solution of 5.0 mmol of diaryltellurium dichloride in dry methanol was added dropwise with constant stirring to a methanolic (~ 5 mL) solution of 5.0 mmol of 1, 2-diaminopropane taken in a round bottom flask. An immediate change in color was observed. The contents were refluxedfor 3-4 h and followed by the addition of saturated methanolic solution of metal salts (2.5 mmol). This resulted in a distinct change in color along with slight precipitation of product. The mixture was then again refluxed for about 8 h and cooled to room temperature. The small amount of precipitated solid was filtered off and the filtrate was concentrated and kept in refrigerator to obtain second crop of crystalline product. This was filtered, washed with petroleum-ether and dried in a vacuum desiccator over P_4O_{10}.

Results and Discussion

$TeCl_4$ when heated with phenol[29], *o*-cresol[30] and anisole[31,32] (R-H) appears to undergo electrophilic substitution reaction where $TeCl_3^+$ unit attacks a position para to the -OH group in the aromatic rings, resulting in the formation of diaryltellurium(IV) dichlorides as per equation:

$$2\,R\text{-}H + TeCl_4 \longrightarrow R_2TeCl_2 + 2\,HCl$$

Diaryltellurium dichlorides when refluxed with 1, 2-diaminopropane in presence of $ZnCl_2$/ $CdCl_2$/ $HgCl_2$ in 2:2:1 molar ratios, yield the desired complexes as shown in Scheme 1. These complexes are crystalline, air stable colored solids and are soluble in polar donor organic solvents. The physical properties and analytical data of these complexes are presented in Table 1.



Where L^1; R = p-hydroxyphenyl, L^2; R = 3-methyl-4hydroxyphenyl, L^3; R p-methoxyphenyl, M = Z(II) Cd(II) and Hg(II)

Scheme 1. Formatoin of the Zn(II), Cd(II) and Hg(II) complexs

Table 1. Analytical data, physical properties and molar conductance for metal complexes

Complex	Empirical formula (Formula weight)	Color	M.P.°C	Yield %	Analysis found (calculated),%						$\wedge_M$at *ca*.10^{-3}M Scm^{2}mol^{-1}
					C	H	N	Cl	Te	M	
ZnL^1Cl_2	$C_{30}H_{36}Cl_2N_4O_4Te_2Zn$ (908.15)	Light pink	180-182*	76	39.35 (39.68)	3.77 (4.00)	5.83 (6.17)	7.41 (7.81)	28.47 (28.10)	7.01 (7.20)	65.8
ZnL^2Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Zn$ (964.25)	Cream	184-186*	69	42.17 (42.35)	4.78 (4.60)	5.31 (5.81)	7.00 (7.35)	26.14 (26.47)	6.48 (6.78)	52.44
ZnL^3Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Zn$ (964.25)	Pale yellow	128-130	78	42.00 (42.35)	4.12 (4.60)	5.26 (5.81)	7.08 (7.35)	26.19 (26.47)	6.38 (6.78)	43.8
CdL^1Cl_2	$C_{30}H_{36}Cl_2N_4O_4Te_2Cd$ (955.15)	Light pink	188-190*	68	37.36 (37.72)	3.61 (3.80)	5.48 (5.87)	7.15 (7.42)	26.30 (26.72)	11.49 (11.77)	**
CdL^2Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Cd$ (1011.25)	Pink	176-178*	59	40.14 (40.38)	4.55 (4.39)	5.30 (5.54)	6.89 (7.01)	24.99 (25.24)	10.92 (11.12)	**
CdL^3Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Cd$ (1011.25)	Light pink	128-130	69	40.21 (40.38)	4.19 (4.39)	5.29 (5.54)	6.79 (7.01)	25.00 (25.24)	10.89 (11.12)	72.5
HgL^1Cl_2	$C_{30}H_{36}Cl_2N_4O_4Te_2Hg$ (1043.33)	Cream	123-125	58	34.24 (34.54)	3.19 (3.48)	5.13 (5.37)	6.55 (6.80)	24.14 (24.46)	19.01 (19.23)	**
HgL^2Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Hg$ (1099.43)	Light pink	218-220*	62	36.80 (37.14)	3.84 (4.03)	4.90 (5.10)	6.14 (6.45)	22.99 (23.21)	18.01 (18.24)	86.16
HgL^3Cl_2	$C_{34}H_{44}Cl_2N_4O_4Te_2Hg$ (1099.43)	Light yellow	110-112	67	36.82 (37.14)	4.35 (4.03)	4.88 (5.10)	6.24 (6.45)	23.35 (23.21)	18.01 (18.24)	78.8

*$\wedge_M$ reported[31] for 1:1 electrolyte in acetonitrile = 120-160 ohm^{-1} cm^2 mol^{-1}; *decomposition temperature, ** not soluble in acetonitrile*

Conductance studies

Molar conductance, Λ_M values for soluble metal complexes in acetonitrile at *ca.* 10^{-3} M are compiled in Table 1. The Λ_M values predict non electrolyte to weak electrolyte type behavior of these complexes as the observed Λ_M values are much less than those reported by Geary[33] for 1:1 electrolyte and hence suggest their formulation as [$MLCl_2$].

Magnetic and electronic spectral studies

No d-d transitions are observed for these metal complexes consistent with the d^{10} configuration and as expected these complexes are diamagnetic in nature. The electronic absorption spectra of these complexes exhibit a broad and intense band in the region 272-239 nm due to CT transitions[34-36]. This CT band may also be mixed with the n - π^* and π - π^* bands of the ligand[35] which sweeps the near UV portion of electromagnetic radiation and extends in the visible region with diminishing absorbance upto 500 nm. This is responsible for the color of the complexes.

IR spectra

The preliminary identification of the synthesized metal complexes has been obtained from infrared spectra. The main IR bands along with their assignments are presented in Table 2. The spectra of the metal complexes are quite complex and thus an attempt has been made to identify the donor sites by comparing the spectra of the complexes with those of corresponding constituent; 1, 2-diaminopropane and diaryltellurium dichlorides. Absence of bands corresponding to free amino group in the metal complexes and appearance of a new single sharp band in the region 3029-3257 cm^{-1} (sometimes mixed with O-H) assignable to ν_{N-H} vibration[20,36-39], provide an evidence for the skeleton of the macrocyclic moiety. Presence of medium to strong intensity band at ~1600-1667 cm^{-1} and 806-830 cm^{-1} attributed to N-H deformations coupled with N-H out of plane bending vibrations[37] also supports this contention.

Table 2. Important IR data (cm^{-1}) for metal complexes

Complex	ν_{N-H}	N-H def.	N-H out of plane bending	ν_{C-N}	ν_{M-N}	ν_{M-Cl}	ν_{Te-N}
[ZnL^1Cl_2]	3225 m	1610 s	828 s	1169 s	460 m	285 m	417 w
[ZnL^2Cl_2]	3037 mb	1620 m	806 m	1158 m	443 m	289 w	423 w
[ZnL^3Cl_2]	3189 m	1612 s	823 m	1178 m	466 m	284 w	418 w
[CdL^1Cl_2]	3029 m	1613 s	830 m	1169 s	443 w	287 m	419 w
[CdL^2Cl_2]	3241 m	1615 m	806 m	1157 m	460 m	280 w	421 w
[CdL^3Cl_2]	3204 m	1625 m	816 m	1161 m	454 m	285 w	416 w
[HgL^1Cl_2]	3257m	1615 m	825 m	1171 m	463 m	286 w	419 w
[HgL^2Cl_2]	3037 m	1610 m	806 m	1158 m	456 m	281 w	415 w
[HgL^3Cl_2]	3180 m	1610 m	818 m	1163 m	452 w	280 w	417 w

(m= medium, b= broad, s= strong, w= weak)

The occurrence of medium to weak intensity bands at ~1173 cm^{-1} and 443-466 cm^{-1} may be assigned to C-N stretching vibration[20,40,41] and M-N stretching[36,42,43] respectively. Presence of new weak intensity bands in the complexes near 423- 415 cm^{-1} corresponding to Te-N vibration also support the formation of tellurium containing macrocyclic ring[20,36,44]. Further medium to weak intensity bands in the region 280-289 cm^{-1} may be assigned to ν(M-Cl) vibration[38,45].

Proton NMR spectra

The proton chemical shifts for the metal complexes which are sufficiently soluble in DMSO –d_6 are presented in Table 3. 1, 2-diaminopropane, $H_2N\text{-}CH(CH_3)\text{-}CH_2\text{-}NH_2$, exhibit protons resonance at0.45 (4H), 1.25(3H), 1.67(2H) and 1.83(1H) δppm due to amino, methyl, methylene and methanetriyl/ methine groups, respectively[46,47]. The synthesized metal complexes do not show any signal due to free amino group. Instead, a broad singlet at 2.12-2.15 δ ppm assignable to coordinated secondary amino group[22,23,48] confirms the formation of macrocyclic skeleton.

Table 3. ^{1}H NMR spectral data (δ ppm) for the 10-membered macrocyclic metal complexes in DMSO-*d*6

Complex	Chemical shift, δ ppm
$[ZnL^1Cl_2]$	2.12 (s, 4H, -NH-), 1.23 (d, 6H, aliphatic $–CH_3$) 2.87-3.30 (m, 6H, $-CH_2$-, -CH- propylenic chain), 6.74^d & $7.64^{\,d}$ (16H , phenyl), 7.82 (s, 4H,OH)
$[ZnL^2Cl_2]$	2.13 (s, 4H, -NH-), 1.24 (d, 6H, aliphatic $–CH_3$) 2.95-3.08 (m, 6H, $-CH_2$-, -CH- propylenic chain), 2.54* (s, 12H, Ar-CH_3), 6.75^d, 7.66^d & 7.72^s (12H, phenyl) 7.51 (s, 4H,OH)
$[ZnL^3Cl_2]$	2.13 (s, 4H, -NH-), 1.22 (d, 6H, aliphatic $–CH_3$) 2.88-3.30 (m, 6H, $-CH_2$-, -CH- propylenic chain), 3.34 (s, 12H, $-OCH_3$), 6.92^d & 7.85^d (16H, phenyl), 9.68 (s, 4H,OH)
$[CdL^1Cl_2]$	2.12 (s, 4H, -NH-), 1.24 (d, 6H, aliphatic $–CH_3$) 2.91-3.34 (m, 6H, $-CH_2$-, -CH- propylenic chain), 6.85^d & 7.38^d (16H , phenyl), 7.80 (s, 4H,OH)
$[CdL^2Cl_2]$**	2.15 (s, 4H, -NH-), 1.29 (d, 6H, aliphatic $–CH_3$) 2.97-3.09 (m, 6H, $-CH_2$-, -CH- propylenic chain), 2.55* (s, 12H, Ar -CH_3), 8.11 (s, 4H,OH)
$[CdL^3Cl_2]$**	1.25 (d, 6H, aliphatic $–CH_3$) 2.96-3.09 (m, 6H, $-CH_2$-, -CH- propylenic chain), 3.79 (s, 12H, $-OCH_3$)
$[HgL^1Cl_2]$	2.14 (s, 4H, -NH-), 1.23 (d, 6H, aliphatic $–CH_3$) 2.92-3.39 (m, 6H, $-CH_2$-, -CH- propylenic chain), 6.92^d & 7.38^d (16H , phenyl), 7.82 (s, 4H,OH)
$[HgL^2Cl_2]$	1.98 (s, 4H, -NH-), 1.26 (d, 6H, aliphatic $–CH_3$) 2.93-3.3 (m, 6H, $-CH_2$-, -CH- propylenic chain), 2.53 (s, 12H, Ar -CH_3), 6.97^d, 7.15^d & 7.31^s (12H, phenyl), 8.8 (s, 4H,OH)

(s= singlet, d= doublet, t= triplet, m= multiplet); * may be mixed with solvent peak, ** poorly resolved

The methyl protons of the propylenic chain and that of the phenyl ring resonate at 1.23-1.29 and 2.49-2.55 δ ppm respectively to give a singlet. The signals due to methylene and methanetriyl protons appears slightly downfield as a multiplet at ~2.87 to 3.39 δ ppm thereby confirming deshielding of these protons due to metal nitrogen coordination.

In comparison to the parent diaryltellurium dichlorides[26,27,49], the phenyl protons in the metal complexes resonate at slightly upfield side (6.55-7.92 δ ppm) as 2Cl are replaced by 2 nitrogen atoms causing flow of electron density from nitrogen to tellurium. Further, the independence of chemical shifts of aryl protons on the metal ions, hints at non -involvement of Te atoms of the macrocycle in coordination with the metal ions. Thus, proton NMR studies on these metal complexes also support the formation of 10-membered ditelluratetraaza macrocycles and their tetra dentate ligation behavior as predicted by IR studies.

Thus on the basis of magnetic moment, conductance, IR, proton NMR and electronic spectral studies, a distorted octahedral geometry as proposed may be assigned to these metal complexes as shown in Figure 1.

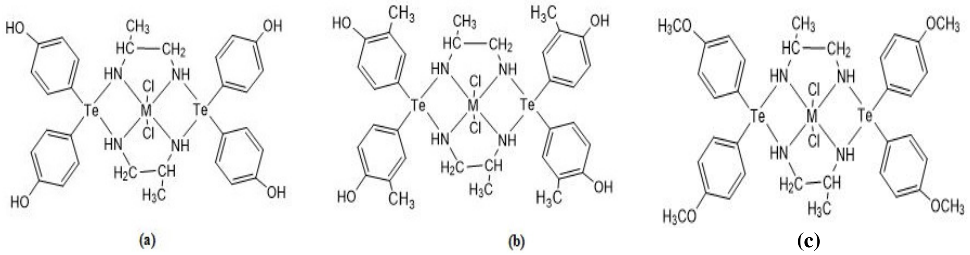


Figure 1. Prosposed structure of the ditellura tetraazamacrocyclic metal complexs of the type $[ML^1Cl_2]$ (a), [ML2Cl2] b and $[ML^3Cl_2]$ (c)

Antimicrobial studies

The MIC values shown by the synthesized metal complexes against the studied fungal and bacterial strains were compared with those of the standard antibiotics cefadroxil and fluconazole (Table 4 and 5). The results have shown that most of the complexes of the tested series possess moderate to good antimicrobial activity as compared to the standard bactericide and fungicide but all the complexes are more active than their precursors against the opportunistic bacteria and fungi and thus indicating that complexation to metal enhances the activity of the ligand. However in the whole series, based on the MIC values, $[HgL^1Cl_2]$, $[HgL^2Cl_2]$ and $[HgL^3Cl_2]$ complexes (MIC= 6.25-12.5 µg/mL) are found to be comparatively more powerful and potent bacteriostatic and fungistatic agent.

Table 4. Minimum inhibitory concentration (MIC) of metal complexes against test bacteria

Complex	MIC(µg mL^{-1})				
	S. aureus	*B. Subtilis*	*E. coli*	*B. cereus*	*S. typhi*
R_2TeCl_2 (*p-hydroxyphenyl*)	25	25	25	25	25
R_2TeCl_2 (3-methyl-4-hydroxyphenyl)	25	25	12.5	25	25
R_2TeCl_2 (*p-methooxyphenyl*)	12.5	25	25	25	25
1,2-diaminopropane	25	25	25	12.5	25
$[ZnL^1Cl_2]$	12.5	12.5	25	12.5	12.5
$[ZnL^2Cl_2]$	12.5	12.5	12.5	12.5	25
$[ZnL^3Cl_2]$	12.5	12.5	12.5	12.5	12.5
$[CdL^1Cl_2]$	25	12.5	12.5	25	25
$[CdL^2Cl_2]$	25	12.5	25	12.5	25
$[CdL^3Cl_2]$	12.5	12.5	12.5	12.5	12.5
$[HgL^1Cl_2]$	6.25	6.25	6.25	6.25	12.5
$[HgL^2Cl_2]$	6.25	6.25	12.5	6.25	12.5
$[HgL^3Cl_2]$	6.25	6.25	12.5	6.25	6.25
Cefadroxil	3.12	1.56	3.12	1.56	3.12

Table 5: Minimum inhibitory concentration (MIC) of metal complexes against test fungi

Complex	MIC, µg mL^{-1}		
	A.niger	*A. fumigatus*	*C.albicans*
R_2TeCl_2 (*p-hydroxyphenyl*)	25	25	25
R_2TeCl_2 (3-methyl-4-hydroxyphenyl)	25	25	12.5
R_2TeCl_2 (*p-methooxyphenyl*)	25	25	25
1,2-diaminopropane	25	25	25
$[ZnL^1Cl_2]$	25	12.5	12.5
$[ZnL^2Cl_2]$	25	25	12.5
$[ZnL^3Cl_2]$	12.5	12.5	12.5
$[CdL^1Cl_2]$	25	12.5	12.5
$[CdL^2Cl_2]$	25	12.5	12.5
$[CdL^3Cl_2]$	12.5	12.5	12.5
$[HgL^1Cl_2]$	12.5	6.25	6.25
$[HgL^2Cl_2]$	6.25	6.25	6.25
$[HgL^3Cl_2]$	6.25	12.5	6.25
Fluconazole	3.12	3.12	1.56

Conclusion

Zn(II), Cd(II) and Hg(II) complexes have been prepared by template condensation of diaryltellurium(IV) dichlorides with 1,2-diaminopropane in presence of divalent metal chlorides. All the metal complexes have been assigned a distorted octahedral geometry on the basis of elemental analyses, conductance measurements, electronicabsorption, IR and proton magnetic resonance spectral studies.Some of the complexes show very good antimicrobial activity against some pathogenic bacteria and fungi while others are moderately active.

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