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Synthesis, Characterization and Antimicrobial Studies on Some Salicylidene-sulphamethoxazole Schiff Base Tellurium (IV) Complexes

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ABSTRACT

A bidentate Schiff base Salicylidene-sulphamethoxazole synthesized from sulphamethoxazole and salicyladehyde, form stable complexes with organyltellurium(IV) trichlorides and diorganryltellurium(IV) dichlorides of the type Sal-SMZ.$RTeCl_2$ and Sal-SMZ.R_2TeCl (where R ***= 4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl and Sal-SMZ = Schiff base)***. Their structures were investigated by elemental analyses, molar conductance, FT-IR and ^1H NMR spectroscopy. The spectral studies predict the coordination sites as phenolic oxygen of Schiff base after deprotonation and nitrogen of the azomethine group, thus giving pentacoordinated tellurium(IV) complexes. The complexes have also been screened for their antifungal and antibacterial activities.

Keywords: Salicylaldehyde, Sulphamethoxazole, Schiff bases, Organyltellurium(IV), Diorganyltellurium(IV), Antifungal and Antibacterial Activities.

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INTRODUCTION

Sulphamethoxazole(SMZ) is an important sulphonamide bacteriostatis antibiotic [1,2] Metal chelates of sulpha drugs have been found to be more bacteriostatic than the drug themselves [3-7]. Schiff bases have shown to exhibit a wide range of biological activities [8-12], industrial applications [13] and in photostabilzation of polymers [14-19]. Medicinal chemists have reported some new derivatives of SMZ including the Schiff base derived salicylaldehyde [20-22]. Several metal complexes of SMZ and its derivatives [23-26] have been reported in literature having functional groups with nitrogen and oxygen donor atom.

Also, aryltellurium(IV) trichlorides are known [27-40] to behave as lewis acids and form complexes with N- , O- and S- donor bases. The diaryltellurium(IV) dichlorides also form such complexes but only with strong chelating ligands [41-43]. In view of this, we have synthesized, characterized and studied antimicriobial activity of some new complexes of salicylidene-sulphamethoxazole Schiff base (Sal-SMZ) with tellurium(IV).

EXPERIMENTAL

Materials and Methods

All preparations were carried out under an atmosphere of dry nitrogen and the solvents used were purified by standard method [44, 45] before use. The purity of compounds was checked by TLC using Silica gel-*G* (Merck). Melting points were determined in open capillary tube and are uncorrected.

Carbon, hydrogen and nitrogen analyses were obtained microanalytically on a Perkin Elmer 2400 Elemental analyser from SAIF, Panjab University Chandigarh. Conductivity was measured in DMSO at 25±2 ˚C with a highly sensitity Systronics conductivity bridge type 306.

FT-IR Spectra were recorded in KBr pellets on a F.T. Infra-Red Spectrophotometer Model RZX (Perkin Elmer) at SAIF, Panjab University Chandigarh. ^{1}H NMR Spectra were recorded in DMSO-d_6 using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer. The antimicrobial screening was carried out by Tube Dilution method at Department of Pharmaceutical Sciences, M. D. University, Rohtak.

Preparation of Organyltellurium(IV) Trichlorides and Diorganyltellurium(IV) Dichlorides

4-Methoxyphenyltellurium(IV) trichloride [46,47], bis(4-methoxyphenyl)tellurium(IV) dichloride [47,48], 4-ethoxyphenyltellurium(IV) trichloride [49], bis(4-ethoxyphenyl)tellurium dichloride[49], 4-hydroxyphenyltellurium(IV) trichloride [50], bis(4-hydroxyphenyl)tellurium(IV) dichloride [50],***3-methyl-4-hydroxyphenyl***tellurium(IV) trichloride [51] and bis(***3-methyl-4-hydroxypheny***l)tellurium(IV) dichloride [51] were prepared by the reactions of $TeCl_4$ with anisole/phenetole/phenol/ *o*-cresol as reported in the literature [46-51].

Preparation of Salicylidene-sulphamethoxazole (Sal-SMZ) Schiff base

Saturated solution of sulphamethoxazole (2 mmol, 0.507 g) in methanol was mixed with salicylaldehyde (2 mmol, 0.212 g) dissolved in 25 mL methanol and 0.1 % methanolic KOH was added to adjust the pH of the solution between 7-8. The reaction mixture was refluxed for 12 h. A clear pale yellow coloured solution was obtained. The Schiff base ligand was isolated by crystallization after volume reduction by evaporation. The crystalline products were dried and kept in a desiccator till further use. Yield = 65 %, M. Pt. = 195- 197˚C.
Analyses (calculated %) $C_{17}H_{15}N_3O_4S$: C (57.15), H (4.20) and N (11.76)
Found: C 56.99, H 3.97 and N 11.47.

Preparation of Salicylidene-sulphamethoxazole complexes of Organyltellurium(IV) trichlorides and Diorganyltellurium(IV) dichlorides

Organyltellurium(IV) trichlorides, $RTeCl_3$ and diorganyltellurium(IV) dichlorides R_2TeCl_2 (R = 4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl***),*** when reacted with salicylidene-sulphamethoxazole in equimolar ratio, yield Sal-SMZ.$RTeCl_2$ and Sal-SMZ.R_2TeCl type complexes.

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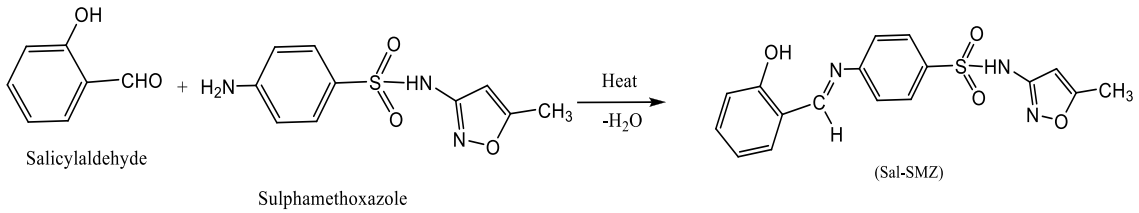
Sodium salt of the ligand was prepared by reacting equimolar (1:1) quantity of sodium metal and Schiff base in methanol. The solvent was distilled off to obtain sodium salt of Schiff base. Then a methanolic saturated solution of 2 mmol of organyltellurium(IV) trichloride or diorganyltellurium(IV) dichloride was added dropwise to suspension of 2 mmol of sodium salt of Schiff base in about 50 mL benzene under reflux. The reaction mixture was further refluxed for 3-4 hours, cooled and precipitated sodium chloride was filtered off. The filterate was then concentrated to about one third of original volume under reduced pressure and cooled in an ice bath to obtain coloured product. This was filtered, washed with benzene + methanol (1:1) and dried in a vacuum desiccators over P_4O_{10}.

RESULTS AND DISCUSSION

Anisole [46-48], phenetole [49], phenol [50] ***and*** *o*-cresol [51] ***undergo*** Friedel-Crafts type reactions with tellurium tetrachloride in boiling organic solvents to form organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides. This reaction involves the electrophilic substitution of the aromatic ring by a trichlorotellurium group at a position *para* to the methoxy/ethoxy/hydroxyl groups.

$$\boldsymbol{R\text{-}H} + TeCl_4 \longrightarrow \boldsymbol{RTeCl_3} + HCl$$
$$\boldsymbol{2\,R\text{-}H} + TeCl_4 \longrightarrow \boldsymbol{R_2TeCl_2} + \boldsymbol{2}\,HCl$$

Preparation of salicylidenesulphamethoxazole Schiff base (Sal-SMZ), by the reaction of sulphamethoxazole and salicylaldehyde can be represented by following equation.



Salicylidene-sulphamethoxazole Schiff base (Sal-SMZ)-sodium salt reacts with organyl tellurium ***(IV) trichlorides and*** diorganyltellurium***(IV) dichlorides in 1:1 molar ratio to yield the corresponding*** aryltellurium***(IV)*** complexes.

$$\text{Sal-SMZ} + RTeCl_3 \xrightarrow[\text{-NaCl}]{Na/CH_3OH} (\text{Sal-SMZ}).RTeCl_2$$

$$\text{Sal-SMZ} + R_2TeCl_2 \xrightarrow[\text{-NaCl}]{Na/CH_3OH} (\text{Sal-SMZ}).R_2TeCl$$

All the tellurium(IV) complexes are coloured, crystalline solids, stable at room temperature and non-hygroscopic in nature. The analytical data and physical properties of ligand and the complexes are recorded in Table 1.

Conductance Studies

Molar conductance (Λ_M) data for the complexes in DMSO are complied in Table 1. Molar conductance, Λ_M data at *ca.* 10^{-3} M for organyltellurium(IV) complexes in DMSO lie in the range 8.80- 51.36 S cm^2 mol^{-1} which predict the weak to 1:1 electrolyte [52,53] type behaviour of these complexes in DMSO, probably due to ionization into RTeCl.Sal-SMZ^+/ R_2Te.Sal-SMZ^+ and Cl^- in DMSO. This conductance behavoiur of tellurium (IV) salicylidene-sulphamethoxazole Schiff base complexes is different from those of transition metal complexes [23], which are reported to be non- electrolytes.

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Table1: Analytical Data, Molar Conductance and Physical Properties of Salicylidene-sulphamethoxazole Schiff Base and Complexes

Compound	Complex (R)	Empirical Formula (Formula Wt.)	Colour (Yield, %)	M. Pt. (°C) dec.	Analyses % Found (Calculated)					Λ_M at *ca.* 10^{-3} M S cm^2mol^{-} in DMSO
					C	H	N	Te	Cl	
Schiff Base	Sal-SMZ	$C_{17}H_{15}N_3O_4S$ (357.27)	Pale yellow (65)	195-197	56.99 (57.15)	3.97 (4.20)	11.47 (11.76)	-	-	-
I	Sal-SMZ.RTeCl_2 (4-methoxyphenyl)	$C_{24}H_{21}Cl_2N_3O_5STe$ (661.85)	Brown (70)	186-188	43.05 (43.55)	2.97 (3.17)	6.01 (6.35)	19.01 (19.28)	10.39 (10.71)	17.37
II	Sal-SMZ.RTeCl_2 (4-ethoxyphenyl)	$C_{25}H_{23}Cl_2N_3O_5STe$ (675.86)	Light brown (65)	173-175	43.99 (44.43)	3.51 (3.40)	5.97 (6.22)	18.51 (18.88)	10.19 (10.49)	20.85
III	Sal-SMZ.RTeCl_2 (4-hydroxyphenyl)	$C_{23}H_{19}Cl_2N_3O_5STe$ (647.84)	Dark brown (75)	170-172	42.41 (42.64)	2.52 (2.93)	6.12 (6.49)	19.47 (19.70)	10.57 (10.94)	29.59
IV	Sal-SMZ.RTeCl_2 (3-methyl-4-hydroxyphenyl)	$C_{24}H_{21}Cl_2N_3O_5STe$ (661.85)	Light orange (82)	158-160	43.14 (43.55)	3.01 (3.17)	6.22 (6.35)	18.99 (19.28)	10.53 (10.71)	11.54
V	Sal-SMZ.R_2TeCl (4-methoxyphenyl)	$C_{31}H_{28}ClN_3O_6STe$ (733.48)	Yellow (66)	178-180	50.37 (50.76)	3.33 (3.82)	5.34 (5.73)	17.09 (17.40)	4.52 (4.83)	42.92
VI	Sal-SMZ.R_2TeCl (4-ethoxyphenyl)	$C_{33}H_{32}ClN_3O_6STe$ (761.50)	Cream (62)	168-170	51.85 (52.05)	4.34 (4.20)	5.23 (5.52)	16.51 (16.76)	4.42 (4.66)	18.10
VII	Sal-SMZ.R_2TeCl (4-hydroxyphenyl)	$C_{29}H_{24}ClN_3O_6STe$ (705.45)	Light yellow (72)	196-198	49.14 (49.38)	3.23 (3.40)	5.47 (5.96)	17.83 (18.09)	4.79 (5.02)	8.80
VIII	Sal-SMZ.R_2TeCl (3-methyl-4-hydroxyphenyl)	$C_{31}H_{28}ClN_3O_6STe$ (733.48)	Light brown (55)	200-202	50.45 (50.76)	3.99 (3.82)	5.57 (5.73)	17.16 (17.40)	4.63 (4.83)	51.36

Values of Λ_M reported [52, 53] for 1:1 electrolytes in DMSO = 50 – 70 S cm^2 mol^{-1}

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Infrared Spectra

The IR data of Schiff base and its tellurium (IV) complexes are listed in Table 2. The infrared spectra of the complexes were compared with those of the free ligand in order to identity the coordination sites.

Table 2: Important IR Data (cm^{-1}) of Schiff Base and Complexes

Compound	$\nu_{(O-H)}$	$\nu_{(C-O)}$	$\nu_{(N-H)}$	$\nu_{(C=N)}$ imine	ν_{C-N} ring	$\nu_{(Te-N)}$	$\nu_{(Te-O)}$
Sal-SMZ	3430 mb	1279 s	3250 w	1650 m	1616 m	-	
I	-	1282 m	3238 w	1632 w	1617 vs	280 w	420 w
II	-	1296 m	3240 w	1638 w	1612 m	292 w	417 w
III	3409 m*	1282 m	3241 m	1628 vw	1615 vs	272 w	412 w
IV	3385 m*	1281 m	3235 w	1630 w	1617 vs	276 w	404 w
V	-	1296 s	3240 vw	1635 w	1618 vs	277 w	406 w
VI	-	1283 s	3238 vw	1628 w	1617 vs	292 w	410 w
VII	3390 w*	1281 m	3232 vw	1634 w	1617 vs	290 w	410 w
VIII	3401 w*	1285 m	3234 vw	1631 w	1618 vs	288 w	415 w

s = strong, vs = very strong, m = medium, w = weak, b = broad.
* Due to phenolic OH of RTe and R_2Te moieties

The $\nu_{(C=N)}$ frequency appearing at 1658 cm^{-1} in free ligand shifts to lower side by about 20-30 cm^{-1} in the complexes, indicating coordination through the azomethine nitrogen [54,55]. Also, $\nu_{(C-O)}$ (phenolic) appearing at around 1279 cm^{-1} in free ligand shifts to 5- 15 cm^{-1} higher frequency in the complexes. This shifting to higher frequency is expected due to maintenance of ring currents arising from the electron delocalization in the chelate ring [24, 56]. The bands for $\nu_{(Te-O)}$ mode [57] appeared in the range of 270-295 cm^{-1} and for $\nu_{(Te-N)}$ mode the bands in the region of 404- 420 cm^{-1} [57] further supports the involvement of phenolic oxygen (after deprotonation) and azomethine nitrogen atom of Schiff base in the coordination. Futher, the non involvement of ring nitrogen and NH nitrogen of Sal-SMZ is confirmed by the presence of $\nu_{C=N}$(imine) at round 1617 cm^{-1} and $\nu_{(N-H)}$ at around 3240 cm^{-1} against 1616 cm^{-1} and 3250 cm^{-1} in the parent ligand, respectively.

Thus, IR data predict the bidentate nature of Sal- SMZ involving azomethine nitrogen atom and phenolic oxygen after deprotonation giving rise to a six membered chelate with the tellurium centre.

^{1}H NMR Spectra

The chemical shift data for the free ligand and complexes are compiled in Table 3. The ^{1}H NMR spectra of the ligand has the expected characteristic signals [22]. The signals at 2.313(singlet), 6.025(singlet), 6.589-7.959(multiplet), 8.938(singlet), 10.245(singlet) and 10.718(singlet) δ ppm suggested the attribution of the protons of the CH_3 group, CH of the isoxazole ring, protons of two aromatic benzene rings, CH=N group, proton of the NH group and proton of the OH group, respectively.

Proton magnetic resonance spectra of these compounds are very complex and a lot of mixing of aryl proton signals of Schiff base and organyltellurium moiety takes place thus making the independent assignment almost impossible. The peak at 10.718 δ ppm in case of ligand disappears in the complexes indicating the involvement of phenolic oxygen in the coordination *via* deprotonation [58].

The azomethine protons resonate as a singlet at 8.891-8.963 δ ppm after coordination through nitrogen atom [59]. Thus salicylidene-sulphamethoxazole ligand acts as a bidentate *ON* ligand in Sal-SMZ.$RTeCl_2$ and Sal-SMZ.R_2TeCl complexes giving penta coordinated tellurium in these complexes as predicated from IR studies as well. The proposed structures are shown in figure 1.

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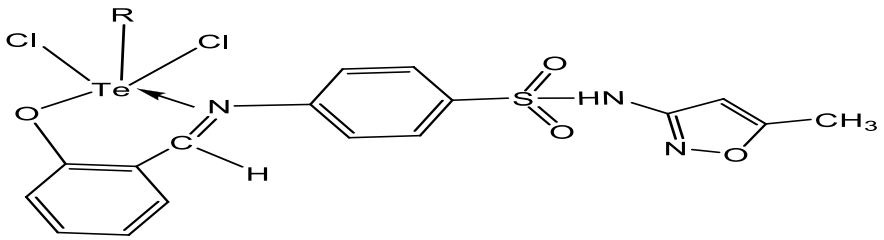
Table 3: ^{1}H NMR Spectral Data of Schiff Base and Complexes in DMSO-d_6

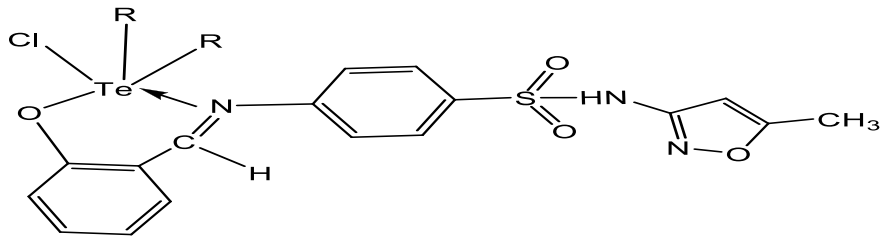
Compound	Chemical Shift, δ ppm
Sal-SMZ	2.313(s, 3H, CH_3), 6.025(s, 1H, isoxazole proton), 6.589-7.959(m, 8H, aryl protons of ring), 8.938(s, 1H, imine proton), 10.24(s, 1H, NH), 10.72(s, 1H, Schiff base OH)
I	2.311(s, 3H, CH_3), 3.815(s, 3H, -OCH_3), 6.081(s, 1H, isoxazole proton), 6.593-7.948(m, 12H, aryl protons of Schiff base and RTe), 8.927(s, 1H, imine proton), 10.224(s, 1H, NH)
II	1.334(t, 3H, -OCH_2**CH_3**), 4.087(q, 2H, -O**CH_2**CH_3), 2.306(s, 3H, CH_3), 6.104 (s, 1H, isoxazole proton), 6.593-7.953(m, 12H, aryl protons of Schiff base and RTe), 8.960(s, 1H, imine proton), 10.269(s, 1H, NH)
III	2.306(s, 3H, CH_3), 6.082(s, 1H, isoxazole proton), 6.605-7.955 (m, 12H, aryl protons of Schiff base and RTe), 8.89(s, 1H, imine proton), 10.20(s, 1H, NH), 10.708(s, 1H, phenolic OH of RTe)
IV	2.312(m, 6H, CH_3), 6.077 (s, 1H, isoxazole proton), 6.589-7.995 (m, 11H, aryl protons of Schiff base and RTe), 8.924(s, 1H, imine proton), 10.223(s, 1H, NH), 10.690(s, 1H, phenolic OH of RTe)
V	2.304(s, 3H, CH_3), 3.792(s, 6H, -OCH_3), 6.027(s, 1H, isoxazole proton), 6.60-7.92(m, 16H, aryl protons of Schiff base and R_2Te), 8.950(s, 1H, imine proton), 10.253(s, 1H, NH)
VI	1.343(t, 6H, -OCH_2**CH_3**), 4.058(q, 4H, -O**CH_2**CH_3), 2.302(s, 3H, CH_3), 6.162(s, 1H, isoxazole proton), 6.600-7.954(m, 16H, aryl protons of Schiff base and R_2Te), 8.933(s, 1H, imine proton), 10.243(s, 1H, NH)
VII	2.310(s, 3H, CH_3), 6.106(s, 1H, isoxazole proton), 6.603-7.956(m, 16H, aryl protons of Schiff base and R_2Te), 8.913(s, 1H, imine proton), 10.223(s, 1H, NH), 10.69(s, 2H, phenolic OH of R_2Te)
VIII	2.129(m, 9H, CH_3), 6.580 (s, 1H, isoxazole proton), 6.965-7.791(m, 14H, aryl protons of Schiff base and R_2Te), 8.963(s, 1H, imine proton), 10.312(s, 1H, NH), 10.981(s, 2H, phenolic OH of R_2Te)

.

s = singlet, d = doublet, q = quartet, t = triplet, m = multiplet



Sal-SMZ.RTeCl$_2$



Sal-SMZ.R$_2$TeCl

Figure 1: Proposed Structures of Complexes

Antimicrobial Activity

The salicylidene-sulphamethoxazole Schiff base (Sal-SMZ) and newly synthesized complexes were screened for their *in vitro* antimicrobial potential against Gram positive bacteria: *Staphylococcus aureus* MTCC 3160 and *Bacillus cereus* MTCC 441, Gram negative bacteria *Escherichia coli* MTCC 443; fungal strains *Aspergillus niger* MTCC 281*, Candida albicans* MTCC 227 by tube dilution method [60]. Dilution of test and standard compounds were prepared double strength nutrient broth- I.P (Antibacterial) and Sabouraud Dextrose Broth –I.P (Antifungal) [61]. This procedure involved preparing two-fold dilutions of compounds (50, 25, 12.5, 6.25, 3.125 μg/mL) in a liquid growth medium dispensed in test tubes. The drug containing tubes were inoculated with a standard bacterial strains and fungal strains. The tubes with bacterial strain were incubated for 24 h at 37˚C whereas the fungal strain tubes were incubated for 7 days at 25±2˚C, the tube were examined for visible bacterial and fungal growth as evidenced by turbidity and results were recorded in terms of MIC (The lowest concentration of test substances which inhibited values are presented in the Table 4.

Table 4: Minimum Inhibitory Concentration MIC (µg/mL) of Schiff base and Complexes

Compound	Bacterial strain			Fungal strain	
	S. aureus MTCC 3160	***B. subtilis*** MTCC 441	***E. coli*** MTCC 443	***A. niger*** MTCC 281	***C. albicans*** MTCC 227
Sal-SMZ	12.5	12.5	6.25	12.5	12.5
I	**3.12**	6.25	6.25	6.25	6.25
II	**3.12**	**3.12**	**3.12**	6.25	**3.12**
III	6.25	**3.12**	**3.12**	**3.12**	6.25
IV	**3.12**	6.25	6.25	12.5	6.25
V	12.5	12.5	12.5	12.5	6.25
VI	12.5	25	12.5	25	12.5
VII	25	12.5	12.5	6.25	12.5
VIII	12.5	12.5	12.5	12.5	6.25

It has been observed that compound I, II,III and IV possess more antibacterial activity against *S. aureus* and *B. cereus* and antifungal data indicate that compound II and III possess better activity against *A. niger* and *C. albicans*.

CONCLUSION

The salicylidene-sulphamethoxazole Schiff base when reacted with organyltellurium chlorides, form two types of complexes: Sal-SMZ.$RTeCl_2$ and Sal-SMZ.R_2TeCl (where R= 4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl). The synthesized complexes were characterized by elemental analyses, conductance measurement, IR and ^{1}H NMR spectral studies. The Schiff base behaves as a uninegative bidentate ligand binding to the tellurium atom via phenolic oxygen after deprotonation and nitrogen of azomethine group, thus forming five membered chelate complexes. The complexes have been observed to possess appreciable antimicrobial activity against bacterial and fungal strains.

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