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

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ABSTRACT

A monobasic bidentate Schiff base Pyridoxylidene-sulphamethoxazole (HPL-SMZ) synthesized from sulphamethoxazole and pyridoxal, form complexes with aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides of the type PL-SMZ.RTeCl$_2$ and PL-SMZ.R$_2$TeCl (R=4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl). They have been characterized by elemental analyses, molar conductance, Fourier Transform Infrared Spectroscopy (FTIR) and Proton Nuclear Magnetic Resonance (^{1}HNMR) spectroscopy. The spectral studies predict the coordination of tellurium through phenolic oxygen after deprotonation and nitrogen of the azomethine group, thus giving pentacoordinated tellurium (IV) complexes probably in a distorted trigonal bipyramidal environment. The complexes were evaluated for their antifungal and antibacterial activities and observed to be more active as compared to parent Schiff base and aryltellurium chlorides.

Keywords: Pyridoxal, Sulphamethoxazole, Schiff base, Aryltellurium (IV), Diaryltellurium (IV), Antifungal, Antibacterial activity

INTRODUCTION

Sulphamethoxazole (SMZ) is an important sulpha drug which act as a bacteriostatic antibiotic [1,2]. Metal complexes of sulpha drugs have been observed to be more bacteriostatic than the drug themselves [3-7]. Schiff bases have shown to exhibit a wide range of biological activities such as, in the treatment of cancer [8], as antibactericidal agents [9,10], as antivirus agents [11], as a fungicidal agents [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 from aldehydes [20-22]. Several metal complexes of SMZ and its derivatives [23-26] have been reported in the 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 antimicrobial activity of some new complexes of Pyridoxylidene-sulphamethoxazole Schiff base (HPL-SMZ) with tellurium (IV).

MATERIALS AND METHODS

All the chemicals used were of Analytical Reagent Grade. All preparations were carried out under an atmosphere of dry nitrogen. The solvents were purified and dried by standard methods and stored under dry conditions.

Carbon, hydrogen and nitrogen analyses were obtained microanalytically on a Thermo Finnigan CHNS analyser from SAIF, Panjab University Chandigarh. Conductance measurements were carried out on a microprocessor based conductivity bridge type Microsil in Dimethyl Sulfoxide (DMSO) at 25 ± 2°C with a dip type conductivity cell (cell constant=1.017). IR spectra were recorded in KBr pellets on an ALPHA Bruker F.T. Infra-Red Spectrophotometer in the region 4000-400 cm^{-1}. ^{1}H NMR Spectra were recorded on Bruker Avance-II 400 NMR spectrometer in DMSO-d$_6$ using Tetramethylsilane (TMS) as an internal reference at SAIF, Panjab University Chandigarh.

Antimicrobial activity was evaluated in acetone against bacterial strain: *Staphylococcus aureus* ATCC-11632 and *Bacillus cereus* MTCC-7350 (Gram positive), *Escherichia coli* ATCC-35218, *Pseudomonas aeruginosa* ATCC-23564, *Providencia rettgeri* DRDE strain and *Salmonella typhi* ATCC 15499 (Gram negative); fungal strains *Aspergillus niger*, *Aspergillus fumigates* and *Aspergillus flavus* using Macrobroth or Tube dilution method [44]. Dilution of test and standard compounds were prepared double strength nutrient broth-I.P (Antibacterial) and Sabouraud Dextrose Broth–I.P (Antifungal) [45]. This procedure involved preparing two-fold dilutions of compounds (20, 10, 5, 2.5, 1.25 and 0.625 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 ± 1°C, the tube were examined for visible bacterial and fungal growth as evidenced by turbidity.

Preparation of aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides

Aryltellurium (IV) trichlorides, $RTeCl_3$ and diaryltellurium (IV) dichlorides, R_2TeCl_2 (R=4-methoxy-, 4-ethoxy-, 4-hydroxy-, 3-methyl-4-hydroxyphenyl) were prepared by the methods reported in the literature [46-51].

Preparation of Pyridoxylidene-sulphamethoxazole (HPL-SMZ) Schiff base

Saturated solution of sulphamethoxazole (2 mmol, 0.507 g) in methanol was mixed with pyridoxal (2 mmol, 0.334 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 about 1 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=78%, M. Pt.=191-193°C.
Analyses (calculated %) $C_{18}H_{18}N_4O_5S$: C (53.72), H (4.51) and N (13.92), Found: C 53.55, H 4.99 and N 13.85 [21].

Preparation of Pyridoxylidene-sulphamethoxazole complexes of aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides

Aryltellurium (IV) trichlorides, $RTeCl_3$ and diaryltellurium (IV) dichlorides R_2TeCl_2 (R=4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl), when reacted with Pyridoxylidene-sulphamethoxazole in equimolar ratio, yield PL-SMZ.$RTeCl_2$ and PL-SMZ.R_2TeCl type complexes.

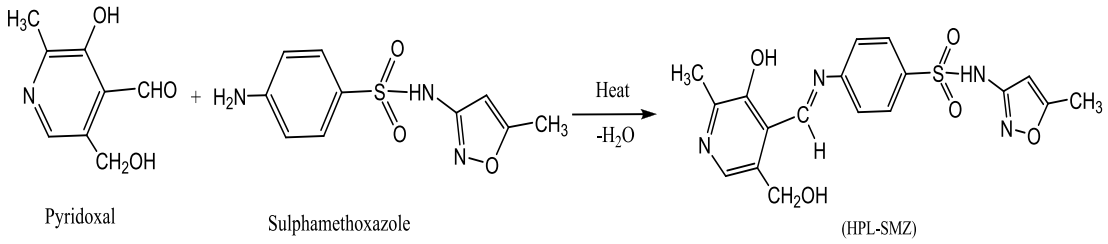
Sodium salt of the ligand was prepared by reacting equimolar (1:1) quantity of sodium metal and Schiff base in methanol. The solvent was distilled off to obtain sodium salt of Schiff base. Then a methanolic saturated solution of 2 mmol of aryltellurium (IV) trichloride or diaryltellurium (IV) dichloride 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 h, cooled and precipitated sodium chloride was filtered off. The filtrate 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 vacuum desiccator 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 aryltellurium (IV) trichlorides and diaryltellurium (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.

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

Preparation of HPL-SMZ, by the reaction of sulphamethoxazole and pyridoxal can be represented by following equation.



Sodium salt of Pyridoxylidene-sulphamethoxazole Schiff base (NaPL-SMZ) reacts with aryl tellurium (IV) trichlorides and diaryltellurium (IV) dichlorides in 1:1 molar ratio to yield the corresponding aryltellurium (IV) complexes.

$$NaPL\text{-}SMZ + RTeCl_3 \longrightarrow (PL\text{-}SMZ).RTeCl_2 + NaCl$$

$$NaPL\text{-}SMZ + R_2TeCl_2 \longrightarrow (PL\text{-}SMZ).R_2TeCl + NaCl$$

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 compiled in Table 1. Molar conductance, Λ_M data at *ca.* 10^{-3} M for aryltellurium(IV) complexes in DMSO lie in the range 33.05-61.32 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.PL-SMZ^+/R_2Te.PL-SMZ^+ and Cl^- in DMSO. This conductance behaviour of tellurium (IV) Pyridoxylidene-sulphamethoxazole Schiff base complexes is different from those of transition metal complexes [23], which are reported to be non-electrolytes.

Infrared spectra

The important 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 identify the coordination sites. Examination of Schiff base spectrum shows the presence of weak band at 2858 cm^{-1} due to intramolecular hydrogen bonding between hydrogen atom of phenolic group of pyridoxal moiety and lone pair on nitrogen atom of azomethine group by forming quasi six membered rings [54-56].

Table 1: Analytical data, molar conductance and physical properties of Pyridoxylidene-sulphamethoxazole Schiff base and tellurium (IV) complexes

Comp.	Complex (R)	Empirical formula (Formula Wt.)	Colour (yield, %)	M. P. (°C)	Analyses % found (Calculated)					Λ_M at *ca.* 10^{-3} M S cm^2 mol^{-1} in DMSO
					C	H	N	Te	Cl	
Schiff Base	HPL-SMZ	$C_{18}H_{18}N_4O_5S$ (402.42)	Pale yellow (78)	191-193	53.55 (53.72)	4.99 (4.51)	13.85 (13.92)	-	-	-
1	PL-SMZ.RTeCl$_2$ (4-methoxyphenyl)	$C_{25}H_{24}Cl_2N_4O_6STe$ (707.05)	Dark yellow (72)	138-140	42.35 (42.47)	3.27 (3.42)	7.52 (7.92)	17.95 (18.05)	9.88 (10.03)	47.59
2	PL-SMZ.RTeCl$_2$ (4-ethoxyphenyl)	$C_{26}H_{26}Cl_2N_4O_6STe$ (721.08)	Light yellow (68)	148-150	43.13 (43.31)	3.51 (3.63)	7.57 (7.77)	17.57 (17.70)	9.59 (9.83)	61.32
3	PL-SMZ.RTeCl$_2$ (4-hydroxyphenyl)	$C_{24}H_{22}Cl_2N_4O_6STe$ (693.03)	Yellow (70)	104-106	41.27 (41.59)	3.01 (3.20)	7.97 (8.08)	18.27 (18.41)	10.19 (10.23)	42.20
4	PL-SMZ.RTeCl$_2$ (3-methyl-4-hydroxyphenyl)	$C_{25}H_{24}Cl_2N_4O_6STe$ (707.05)	Pale yellow (80)	72-74	42.27 (42.47)	3.55 (3.42)	7.63 (7.92)	17.88 (18.05)	9.91 (10.03)	33.86
5	PL-SMZ.R$_2$TeCl (4-methoxyphenyl)	$C_{32}H_{31}ClN_4O_7STe$ (778.73)	Cream (65)	94-96	49.25 (49.36)	3.84 (4.01)	7.03 (7.19)	16.27 (16.39)	4.30 (4.55)	37.01
6	PL-SMZ.R$_2$TeCl (4-ethoxyphenyl)	$C_{34}H_{35}ClN_4O_7STe$ (806.78)	Yellow (60)	124-126	50.52 (50.62)	4.59 (4.37)	5.55 (6.94)	15.53 (15.82)	4.17 (4.39)	38.54
7	PL-SMZ.R$_2$TeCl (4-hydroxyphenyl)	$C_{30}H_{27}ClN_4O_7STe$ (750.68)	Yellowish orange (72)	108-110	47.89 (48.00)	3.51 (3.63)	7.27 (7.46)	16.88 (17.00)	4.60 (4.72)	34.17
8	PL-SMZ.R$_2$TeCl (3-methyl-4-hydroxyphenyl)	$C_{32}H_{31}ClN_4O_7STe$ (778.73)	Dark brown (50)	112-114	49.15 (49.36)	4.13 (4.01)	7.07 (7.19)	16.29 (16.39)	4.35 (4.55)	33.05

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

This band disappears on complexation with tellurium and shows that the phenolic group of pyridoxal moiety is involved in bonding [57] after deprotonation. Also, an intense ligand band at 1260 cm^{-1} (phenolic –C-O) in free ligand has shifted to higher frequency side in complexes, further proved that the phenolic group coordinates to tellurium atom [58,59] after deprotonation.

In addition to this, Schiff base, band at 1604 cm^{-1} due to vibration of azomethine group [22,60,61], acts shifted to higher wave numbers by 20 cm^{-1} [21,62,63] in the complexes indicating thereby the participation of the azomethine nitrogen in coordination [64]. The band at 1464 cm^{-1} assigned to pyridine $\nu_{C=N}$ ring vibration of pyridoxal moiety in Schiff base, does not shift in complexes suggesting that nitrogen of pyridine ring is not involved in coordination to tellurium [65]. The new bands for $\nu_{(Te\text{-}O)}$ mode [66,67] appeared in the range of 281-297 cm^{-1} and for $\nu_{(Te\text{-}N)}$ mode the bands in the region of 414-431 cm^{-1} [68] further support the involvement of phenolic oxygen (after deprotonation) and azomethine nitrogen atom of Schiff base in the coordination.

Thus, IR data predict the bidentate nature of HPL-SMZ involving azomethine nitrogen atom and phenolic oxygen after deprotonation giving rise to pentacoordinated tellurium center with distorted trigonal bipyramidal stereochemistry.

Table 2: Important IR data (cm^{-1}) of Schiff base and complexes

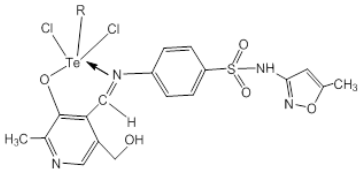
Compound	Phenolic ($\nu_{(O\text{-}H)}$)	Azomethine ($\nu_{(C=N)}$)	Pyridine ring ($\nu_{(C=N)}$)	Phenolic ($\nu_{(C\text{-}O)}$)	Azo-N ($\nu_{(Te\text{-}N)}$)	Phen-O ($\nu_{(Te\text{-}O)}$)
HPL-SMZ	2858 wb	1604** sh	1464 sh	1260 s	-	-
1	-	1615 sh	1462 s	1318 s	429 w	281 w
2	-	1612 sh	1464 s	1310 s	428 w	297 w
3	3206 w*	1618 sh	1465 s	1266 s	423 w	293 w
4	3372 w*	1620 sh	1470 s	1319 s	420 w	286 w
5	-	1614 s	1474 s	1305 s	414 w	291 w
6	-	1617 sh	1464 s	1306 s	430 w	283 w
7	3240 w*	1618 sh	1467 s	1269 s	428 w	296 w
8	3277 w*	1622 sh	1467 s	1321 s	431 w	288 w

s=Sharp, w=Weak; sh=Shoulder; *Due to phenolic OH of RTe and R$_2$Te moieties; **Not well resolved

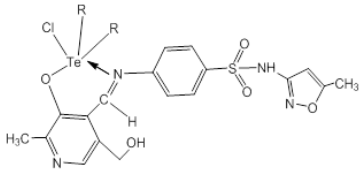
^{1}H NMR spectra

The chemical shift data for the free ligand and complexes are compiled in Table 3. The phenolic proton resonating at 10.801 δ ppm in Schiff base due to presence of intramolecular hydrogen bonding [57,69-71], disappears on complexation indicating the involvement of phenolic oxygen in the coordination after deprotonation [71]. The signal due to methanolic [57] proton is observed at around 3.529 δ ppm which remain intact in complexes suggesting that –CH$_2$OH attached to the pyridoxal ring does not participate in bonding. The azomethine proton [61,71] resonating at 8.114 δ ppm in parent ligand shifts to downfield side in the complexes indicating thereby the deshielding of azomethine proton due to its coordination with tellurium atom [71].

Thus, Pyridoxylidene-sulphamethoxazole ligand acts as a monobasic bidentate *–N, -O* chelating ligand in PL-SMZ.RTeCl$_2$ and PL-SMZ.R$_2$TeCl complexes giving five coordinated tellurium having Ψ-TBP geometry in these complexes as predicated from IR studies as well. The proposed structures are shown in Figure 1.



PL-SMZ.RTeCl$_2$



PL-SMZ.R$_2$TeCl

R= 4-methoxyphenyl, 4-ethoxyphenyl,4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl

Figure 1: Proposed structures of complexes

Table 3: ^{1}HNMR Spectral Data of Schiff Base and complexes in DMSO-d$_6$

Compound	Phenolic-OH	Methanolic-OH	Azomethine-H	Aromatic protons	Isoxazole proton
HPL-SMZ	10.801 s	3.529 s	8.114 s	6.586-7.502 m	6.061 s
1	-	3.477 s	8.269 s	6.578-8.166 m	6.060 s
2	-	3.395 s	8.355 s	6.120-8.118 m	6.086 s
3	10.778* s	3.366 s	8.315 s	6.594-8.011 m	6.096 s
4	10.989* s	3.396 s	8.336 s	6.576-8.212 m	6.064 s
5	-	3.356 s	8.120 s	6.580-7.965 m	6.002 s
6	-	3.592 s	8.155 s	6.580-8.012 m	6.098 s
7	10.832* s	3.348 s	8.190 s	6.588-8.069 m	6.064 s
8	10.969* s	3.207 s	8.263 s	6.785-8.128 m	6.067 s

s=Singlet; m=Multiplet; *Due to phenolic OH of RTe and R$_2$Te moieties

Antimicrobial activity

The Pyridoxylidene-sulphamethoxazole Schiff base and newly synthesized aryltellurium (IV) Schiff base complexes were evaluated for *in vitro* antibacterial and antifungal activity and the results in terms of minimum inhibitory concentration are presented in Table 4. Comparative study of the MIC value of Schiff base (HPL-SMZ) and their tellurium (IV) complexes shows that the complexes exhibit higher antibacterial and antifungal activity than ligand itself. The antimicrobial activity shows following trend:

PL-SMZ.R$_2$TeCl>PL-SMZ.RTeCl$_2$>>Schiff base

It has been observed that all tellurium Schiff base complexes exhibit appreciable activity against *S. aureus* except compound number 2 and compound 3 shows activity only against *P. rettgeri* whereas Schiff base does not show any activity against these strains. In case of antifungal screening, Schiff base is active only against *A. niger* but complexes exhibit moderate activity against all strains.

Table 4: Minimum inhibitory concentration, (µg/ml); (-) resistant

	Bacterial strains						Fungal strains		
Compound	***Staphylococcus aureus***	***Salmonella typhi***	***Pseudomonas aeruginosa***	***Escherichia coli***	***Bacillus cereus***	***Providencia rettgeri***	***Aspergillus niger***	***Aspergillus fumigates***	***Aspergillus flavus***
HPL-SMZ	-	20	10	5	1.25	-	20	-	-
1	1.25	-	5	1.25	0.625	-	5	-	-
2	-	20	10	5	1.25	-	5	10	5
3	5	10	5	20	-	0.625	-	1.25	5
4	1.25	2.5	1.25	5	-	-	-	-	-
5	1.25	2.5	1.25	5	-	-	-	10	-
6	1.25	-	5	1.25	0.625	-	20	5	1.25
7	20	-	1.25	-	0.625	-	20	5	1.25
8	5	-	-	-	1.25	2.5	20	-	-

CONCLUSION

The Pyridoxylidene-sulphamethoxazole Schiff base when reacted with aryltellurium (IV) and diaryltellurium (IV) chlorides form 1:1 type complexes. The synthesized complexes were characterized by elemental analyses, conductance measurement, IR and ^{1}HNMR spectral studies. The Schiff base (HPL-SMZ) in these complexes behaves as a monobasic bidentate ligand binding to the tellurium atom via phenolic oxygen after deprotonation and nitrogen of azomethine group, thus forming penta coordinated tellurium center. The complexes have been observed to possess higher antimicrobial activity against bacterial and fungal strains than the Schiff base.

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