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RESEARCH ARTICLE

N-(2-Pyridyl)-5-chlorosalicylideneimine Schiff Base and its Tellurium(IV) Complexes: Synthesis, Characterization and *In Vitro* Biological Activities

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Abstract: Tridentate Schiff base (2-APY-{5-ClSalH}) has been prepared by the condensation of 5-chlorosalicylaldehyde with 2-aminopyridine and further new tellurium(IV) complexes *i.e.* [$TeCl_3$(2-APY-{5-ClSal})], [$RTeCl_2$(2-APY-{5-ClSal})] and [R_2TeCl(2-APY-{5-ClSal})]: where R = *p*-methoxyphenyl, *p*-hydroxyphenyl, 3-methyl-4-hydroxyphenyl, of this Schiff base have been synthesized**.** The characterization of ligand and complexes was carried out by elemental analyses, conductivity measurements, FT-IR and ^1H NMR spectral data. The Schiff base ligand as well as their some tellurium(IV) complexes were tested for antimicrobial activities *in vitro* against Gram-positive bacteria *i.e. Staphylococcus aureus* and *Streptococcus pyogenes* and Gram-negative bacteria *i.e. Pseudomonas aeruginosa* and *Escherichia coli* and fungi *i.e. Candida albicans*, *Aspergillus niger* and *Aspergillus clavatus* by broth dilution method.

Keywords: 5-Chlorosalicylaldehyde, Tridentate, Schiff base, 2-Aminopyridine, Antimicrobial activities

Introduction

A Schiff base is the nitrogen analogue of aldehyde in which the C=O group is replaced by a C=N group[1]. They also exhibit a variety of biological activities but with substituted salicylaldehyde compounds possesses higher activities[2]. This has led to concentrate deep research on this class of compounds[3] and their metal complexes[4,5]. Similarly, the presence of hetro-atoms in the Schiff bases enhances activity[6], particularly when the group such as –OH / -SH are near to azomethine group, resulting in the formation of five or six membered ring complexes[7-12]. Schiff bases are reported to have biological activities like antibacterial[13-21], antifungal[13,15-18,22], antitumor[16,23,24], antiviral[25-27], anti-HIV[28], herbicidal[29] and anti influenza A virus[30] activities.

Tellurium(IV) chloride is also known to form adducts with amides[31,32] and thiourea[33], thus reflecting its acceptor behaviour[7,8,34-41]. The present study has thrown light on the chelating behaviour of Schiff base derived from 5-chlorosalicylaldehyde and 2-aminopyridine towards organyltellurium(IV) chlorides which are known[7,8,34-58] to act as Lewis acids. These complexes have been examined for their antimicrobial activity against different strains of bacteria and fungi.

Experimental

All chemicals used were of analytical reagent grade. All preparations were carried out under an atmosphere of dry N_2 atmosphere. The solvents were purified by standard method[59,60] 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 from SAIF, Panjab University Chandigarh on a ThermoFinnigan CHNS analyser. Conductance studies were performed under dry condition in DMSO at 25±2 °C with a dip type conductivity cell on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra (4000-40 cm^{-1}) were recorded in KBr and polyethylene pellets for Mid-IR and Far-IR respectively, on a FT-Infra-Red spectrometer Model Nicolet IS50 (Thermo Scientific). Proton NMR Spectra were recorded in DMSO-d_6 using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer.

Preparation of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides

4-Methoxyphenyltellurium(IV) trichloride[61,62], bis(*p*-methoxyphenyl)tellurium(IV) dichloride[62,63], 4-hydroxyphenyltellurium(IV) trichloride[64], bis(*p*-hydroxyphenyl) tellurium(IV) dichloride[64], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride[65] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride[65] were prepared by the reactions of tellurium tetrachloride (Aldrich) with corresponding arenes *i.e.* anisole, phenol, *o*-cresol respectively, by the methods reported in the literature[61-65].

Preparation of 5-chlorosalicylaldehyde-2-aminopyridine (2-APY-{5-ClSalH}) Schiff base

The Schiff base was prepared by mixing equimolecular quantity of 5-chlorosalicylaldehyde (0.08 mole, 12.53 g) and 2-aminopyridine (0.08 mole, 7.52 g) in 25 mL methanol in a round bottomed flask equipped with a condenser[66]. The reaction mixture was refluxed on water bath for 4 hours. After cooling, the precipitated Schiff base was collected by filtration and recrystallized from methanol. The orange shiny crystalline product was dried in a desiccator over anhydrous $CaCl_2$ and kept over P_4O_{10}. Yield = 88%, m.pt. = 123-125 °C.

Preparation of complexes

Tellurium tetrachloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides, when reacted with Schiff base (2-APY-{5-ClSalH}), form solid complexes as described below:

[$TeCl_3$(2-APY-{5-ClSal})], [$RTeCl_2$(2-APY-{5-ClSal})] and [R_2TeCl(2-APY-{5-ClSal})]

The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL anhydrous methanol to a hot solution of 5 mmol Schiff base (2-APY-{5-ClSalH}) in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent was distilled off to obtain the desired products which were recrystallized from dry methanol. The coloured complexes crystallized out, which were filtered, washed with dry methanol and dried in a vacuum desiccator over P_4O_{10}.

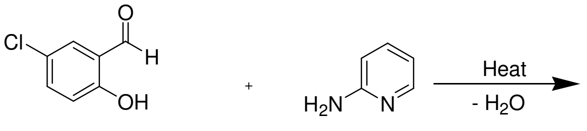
Results and Discussion

Anisole[61-63], phenol[64] and *o*-cresol[65] (R-H) undergo Friedel-Crafts type condensation reaction when reacted 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 / hydroxyl groups.

$$\text{R-H} + TeCl_4 \longrightarrow RTeCl_3 + HCl \quad (1)$$

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

Preparation of Schiff Base by the reaction of 5-chlorosalicylaldehyde with 2-aminopyridine are represented in the following equation.



(3)

5-Chlorosalicylaldehyde 2-Aminopyridine (2-APY-{5-ClSalH})

Schiff Base reacts with tellurium(IV) chloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides in 1:1 molar ratio to yield the corresponding organyltellurium(IV) complexes.

$$TeCl_4 + (\text{2-APY-\{5-ClSalH\}}) \longrightarrow TeCl_3(\text{2-APY-\{5-ClSal\}}) \quad (4)$$

$$RTeCl_3 + (\text{2-APY-\{5-ClSalH\}}) \longrightarrow RTeCl_2(\text{2-APY-\{5-ClSal\}}) \quad (5)$$

$$R_2TeCl_2 + (\text{2-APY-\{5-ClSalH\}}) \longrightarrow R_2TeCl(\text{2-APY-\{5-ClSal\}}) \quad (6)$$

All the tellurium(IV) complexes are colored, crystalline solids, stable at room temperature and non-hygroscopic in nature. They are insoluble in non polar and less polar organic solvents, but are soluble in polar donor solvents like DMF, DMSO *etc*. The analytical data along with their physical properties are presented in Table 1.

Conductance studies

The molar conductance (Λ_M) data for organyltellurium(IV) Schiff base complexes in DMSO are compiled in Table 1. The Λ_M values at *ca.* 10^{-3} M of complexes lies in the range 19.45-53.06 S cm^2 mol^{-1} which predict the non-electrolyte to 1:1 weak electrolyte type behavior[67,68] of these Schiff base complexes in DMSO, probably due to ionization into $TeCl_2$(2-APY-{5-ClSal})$^+$ / RTeCl(2-APY-{5-ClSal})$^+$ / R_2Te(2-APY-{5-ClSal})$^+$ and Cl^- in DMSO. The higher Λ_M values for some complexes may be due to steric factors and donor behavior of DMSO to result in probable dissociation into solvated cation and 2-APY-{5-ClSal}$^-$ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes[69] which are reported to be non-electrolytes.

Infrared spectra

The IR spectra for our studied complexes and free ligand listed in Table 2 give information about the coordination of the ligand to metal. The IR spectrum of the ligand indicate that the $\upsilon_{(C=N)}$ band[31,38,41,66,70] of the ligand at 1613 cm^{-1} is due to the azomethine linkage which were shifted towards lower frequency[22,38,41,66,70-72] indicating that the ligand coordinate to tellurium metal ions via the azomethine nitrogen[73,74].

Table 1. Analytical data, molar conductance and physical properties for Schiff base (2-APY-{5-ClSalH}) complexes of tellurium(IV)

Compd. No.	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^{-1} in DMSO
					C	H	N	Te	Cl	
Schiff Base	(2-APY-{5-ClSalH})	$C_{12}H_9ClN_2O$ (232.64)	Orange (88)	123-125	61.74 (61.95)	3.95 (3.87)	11.92 (12.04)	-	15.14 (15.26)	-
I	$TeCl_3$(2-APY-{5-ClSal})	$C_{12}H_8Cl_4N_2OTe$ (465.74)	Yellowish green (74)	118-120	30.72 (30.94)	1.84 (1.72)	5.89 (6.02)	27.32 (27.40)	30.35 (30.49)	23.51
II	$RTeCl_2$(2-APY-{5-ClSal}) (*p*-methoxyphenyl)	$C_{19}H_{15}Cl_3N_2O_2Te$ (537.31)	Cream (71)	154-156	42.24 (42.47)	2.85 (2.79)	5.13 (5.21)	23.62 (23.75)	19.71 (19.82)	32.11
III	$RTeCl_2$(2-APY-{5-ClSal}) (*p*-hydroxyphenyl)	$C_{18}H_{13}Cl_3N_2O_2Te$ (523.30)	Brown (78)	189-191	41.15 (41.31)	2.56 (2.48)	5.27 (5.35)	24.46 (24.38)	20.22 (20.35)	19.45
IV	$RTeCl_2$(2-APY-{5-ClSal}) (3-methyl-4-hydroxyphenyl)	$C_{19}H_{15}Cl_3N_2O_2Te$ (537.31)	Light Brown (81)	171-173	42.31 (42.47)	2.87 (2.79)	5.08 (5.21)	23.89 (23.75)	19.68 (19.82)	27.09
V	R_2TeCl(2-APY-{5-ClSal}) (*p*-methoxyphenyl)	$C_{26}H_{22}Cl_2N_2O_3Te$ (608.88)	Light Orange (69)	220-222	51.04 (51.28)	3.69 (3.61)	4.47 (4.60)	20.84 (20.96)	11.54 (11.66)	46.87
VI	R_2TeCl(2-APY-{5-ClSal}) (*p*-hydroxyphenyl)	$C_{24}H_{18}Cl_2N_2O_3Te$ (580.86)	Light Brown (79)	173-175	49.47 (49.62)	3.22 (3.10)	4.71 (4.82)	21.82 (21.97)	12.05 (12.22)	53.06
VII	R_2TeCl(2-APY-{5-ClSal}) (3-methyl-4-hydroxyphenyl)	$C_{26}H_{22}Cl_2N_2O_3Te$ (608.88)	Light Grey (85)	163-165	51.09 (51.28)	3.72 (3.61)	4.52 (4.60)	21.08 (20.96)	11.49 (11.66)	50.21

Values of Λ_M reported[67,68] for 1:1 electrolytes in DMSO = 50-70 S cm^2 mol^{-1}

Table 2. Important IR data (cm^{-1}) of the Schiff base (2-APY-{5-ClSalH}) and complexes

Compound No.	(Phenolic) $\nu_{(OH)}$	(Azomethine) $\nu_{(C=N)}$	(Pyridine) $\nu_{(C\text{-}N\text{-}C)}$	$\nu_{(Te\text{-}O)}$	$\nu_{(Te\text{-}N)}$
(2-APY-{5-ClSalH})	3085 b	1613 s	1486 s	-	-
I	-	1568 m	1542 s	293 s	409 s
II	-	1581 s	1542 mb	293 s	420 s
III	-	1580 m	1541 m	290 s	419 s
IV	-	1586 s	1543 s	292 s	416 s
V	-	1569 m	1544 m	292 s	420 s
VI	-	1576 s	1544 m	290 s	419 s
VII	-	1576 s	1544 s	289 s	419 s

s = strong, m = medium, b = broad

The absence of peak due to the phenolic –OH group at 3085 cm^{-1} suggest the coordination of the ligand to the metal via deprotonation. Schiff base coordination through pyridine nitrogen affecting the $\upsilon_{(C\text{-}N\text{-}C)}$ band, therefore the frequency in free ligand at 1486 cm^{-1} was moved to a higher value and appears in the region 1541-1544 cm^{-1} in the spectrum of complexes.

The new strong bands in the spectra of complexes appear in the region 289-293 cm^{-1} and 409-420 cm^{-1} are ascribed to region might be due to $\upsilon_{(Te\text{-}O)}$[38,41,71,72,75,76] and $\upsilon_{(Te\text{-}N)}$[77] stretching and confirms that the phenolic oxygen, azomethine and pyridine nitrogen atoms are the coordination sites of the tridentate ligand.

Thus the IR data infers that in studied complexes each tellurium atom is in octahedral environment is coordinated by tridentate monobasic *ONN* Schiff base, chlorides and organyl groups (in the different numbers).

^{1}H NMR spectra

Further, evidence for the coordination mode of the ligand is obtained by the ^{1}H NMR studies. The ^{1}H NMR spectra data was recorded in DMSO-d$_6$ and presented in Table 3.

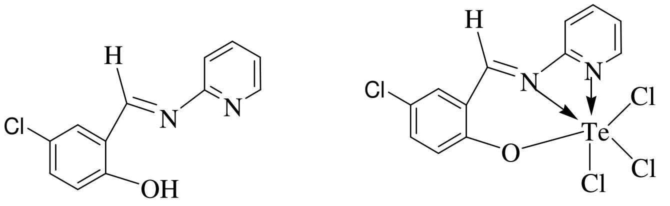
Table 3. ^{1}H NMR spectral data of Schiff base (2-APY-{5-ClSalH}) and complexes

Compound No	(Phenolic) -OH δ ppm	(Azomethine) -HC=N δ ppm	(Ar rings protons) δ ppm	$-CH_3/-OCH_3^*$ δ ppm	-OH of Te/R_2Te δ ppm
(2-APY-{5-ClSalH})	13.460 (s, 1H)	9.392 (s, 1H)	6.970-8.527 (cm, 7H)	-	-
I	-	10.254 (s, 1H)	6.955-8.012 (cm, 7H)	-	-
III	-	10.262 (s, 1H)	7.596-8.651 (cm, 11H)	-	9.025 (s,1H)
V	-	10.265 (s, 1H)	6.840-8.237 (cm, 15H)	3.368 (s,6H*)	-
VI	-	10.271 (s, 1H)	7.587-8.024 (cm, 15H)	-	8.989 (s,2H)
VII	-	10.265 (s, 1H)	6.849-8.101 (cm, 13H)	2.509 (s,6H)	9.014 (s,2H)

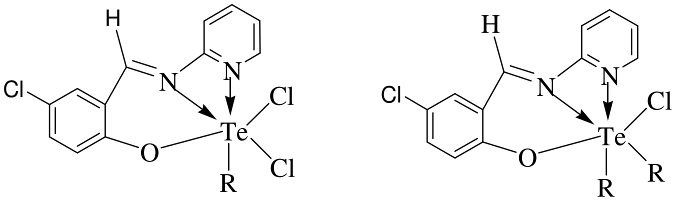
s = singlet, cm = complex multiplet. Spectra of compound numbers II and IV are not well resolved due to poor solubility

The absence of a singlet in high frequency region (δ 13.460 ppm), which typically corresponds to phenolic hydrogen indicates that *N*-2-pyridyl-5-chlorosalicylideneimine binds tellurium by deprotonated phenolic oxygen. The singlet that appears at δ 9.392 ppm is ascribed to azomethine hydrogen, which is less shielded after coordination through azomethine nitrogen[38,41,50,51,70,78] and appear in the downfield region 10.254-10.271 δ ppm. The complex multiplets found in the spectrum of complexes in the δ 6.840-8.651 ppm region corresponds to coupled hydrogen atoms of aromatic rings.

The ^{1}H NMR spectroscopic data are in good agreement with others experimental results and strongly support the proposed structure of the complexes. On the basis of these studies, the proposed structures for the complexes are as below (Figure 1).



(2-APY-{5-ClSalH}) $TeCl_3$(2-APY-{5-ClSal})



$RTeCl_2$(2-APY-{5-ClSal}) R_2TeCl(2-APY-{5-ClSal})

Figure 1. Proposed structures of Schiff base (2-APY-{5-ClSalH}) and tellurium(IV) complexes

Biological studies

In vitro antibacterial activity of the Schiff base (2-APY-{5-ClSalH}) and their tellurium(IV) complexes were evaluated[79] against two Gram-positive bacteria (*S. aureus* MTCC 96 and *S. pyogenes* MTCC 442), two Gram-negative bacteria (*P. aeruginosa* MTCC 1688 and *E. coli* MTCC 443) and antifungal activity against three fungal strains *C. albicans* MTCC 227, *A. niger* MTCC 282 and *A. clavatus* MTCC 1323.

The factors that govern antibacterial activities are strongly dependent on the central metal ion and the coordination numbers and also due to the presence of nitrogen and sulfur donor groups[80-82].

Comparative study of Schiff base and tellurium(IV) complexes indicates that the complexes show higher antibacterial activity than antifungal activity. Schiff base and complex no. III and VI *i.e.* [$RTeCl_2$(2-APY-{5-ClSal})] and [R_2TeCl(2-APY-{5-ClSal})]: where R = *p*-hydroxyphenyl respectively, show substantial activity against two bacterial strains (*P. aeruginosa* and *E. coli*) and even comparable to standard drug *ampicillin*. The data is given in Table 4.

Table 4. Minimum inhibitory concentration MIC (µg/mL) of Schiff base (2-APY-{5-ClSalH}) and complexes

Compound Number	Bacterial Strain				*Fungal Strain*		
	S. aureus MTCC 96	*S. pyogenes* MTCC 442	*P. aeruginosa* MTCC 1688	*E. coli* MTCC 443	*C. albicans* MTCC 227	*A. niger* MTCC 282	*A. clavatus* MTCC 1323
(2-APY-{5-ClSalH})	250	250	100	62.5	500	1000	1000
1	500	500	200	200	**250**	1000	1000
2	250	250	250	250	1000	1000	1000
3	250	250	**100**	**62.5**	1000	1000	1000
6	500	500	**100**	**125**	500	**500**	**500**
Standard Drugs							
Ampicillin	250	100	100	100	-	-	-
Chloramphenicol	50	50	50	50	-	-	-
Nystatin	-	-	-	-	100	100	100
Greseofulvin	-	-	-	-	500	100	100

Conclusion

The complexes of tellurium(IV) with Schiff base derived from 5-chlorosalicylaldehyde and 2-aminopyridine have been synthesized and characterized by elemental analyses, conductance measurement, IR and ^{1}H NMR spectral studies. All the spectral studies suggest that the Schiff base is tridentate in nature having *ONN* donor atoms with distorted octahedral geometry. The complexes have been observed to possess good antimicrobial activity against some bacterial and fungal strains.

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