

Tellurium(IV) Complexes Containing Bidentate Schiff Base and their Biological Activities

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Abstract: Monobasic (*ON*) bidentate Schiff base ligand has been prepared by condensing *o*-hydroxybenzaldehyde with 3-aminopyridine. The new organytellurium(IV) complexes, having formula [TeCl₃(3-APY-{Sal})], [R₂TeCl₂(3-APY-{Sal})] and [R₂TeCl(3-APY-{Sal})] have been synthesized by reacting equimolar ratio of Schiff base (3-APY-{SalH}) with TeCl₄, RTeCl₃ and R₂TeCl₂; where R=4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl. The characterization of Schiff base and its complexes were carried out by elemental analyses, conductivity measurements, FTIR and ¹H NMR spectral studies. Also, the biological activities (antifungal and antibacterial) of the Schiff base ligand and some of its tellurium(IV) complexes have been studied.

Keywords: Schiff base complexes, Monobasic, Bidentate, *o*-Hydroxybenzaldehyde, 3-Aminopyridine.

Introduction

Schiff bases are the condensation product of carbonyl groups (aldehydes or ketones) with amine^{1,2}. Schiff base ligands are considered to be good chelating agents and -OH functional group close to azomethine group made them as special class of ligands³⁻⁶. Some Schiff bases are reported to possess antibacterial^{1,7-12}, antifungal⁹⁻¹³ and antitumor activities^{14,15}. It is reported^{1,16} in the literature that heteroatom increases the Schiff base activity and also Schiff bases derived from substituted *o*-hydroxybenzaldehyde exhibit more antimicrobial activity^{17,18}. Literature survey reveals that a number of metal complexes of Schiff bases derived from 2-aminopyridine and 2,3-diaminopyridine with *o*-hydroxybenzaldehyde, *p*-hydroxybenzaldehyde and *p*-nitrobenzaldehyde *etc.*, have been reported to possess antimicrobial activity¹⁹⁻³⁰.

Tellurium(IV) chloride is also known to form adducts with amides³¹⁻³⁸ and thiourea³⁹, thus reflecting its acceptor behaviour. Also, organytellurium(IV) chlorides are known^{31-36, 40-56} to behave as Lewis acids and form complexes with several N-, O- and S- donor bases. In view of this, we have investigated the reactions of tellurium(IV) chloride and organytellurium(IV) chlorides with *o*-hydroxybenzaldehyde-3-aminopyridine Schiff base (3-APY-{SalH}), to synthesize some new complexes of tellurium(IV).

Experimental

All chemicals used were of analytical reagent grade. All preparations were carried out under an atmosphere of dry N₂ atmosphere. The solvents were purified by standard method^{57,58} 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. Conductivity was measured 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-d₆ using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer from CIL, Guru Jambheshwar University of Science and Technology, Hissar, Haryana, India.

Preparation of organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides

4-Methoxyphenyltellurium(IV) trichloride^{59,60}, bis(*p*-methoxyphenyl)tellurium(IV) dichloride^{60,61}, 4-hydroxyphenyltellurium(IV) trichloride⁶², bis(*p*-hydroxyphenyl) tellurium(IV) dichloride⁶², 3-methyl-4-hydroxyphenyltellurium(IV) trichloride⁶³ and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride⁶³ 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 literature⁵⁹⁻⁶³.

Preparation of o-Hydroxybenzaldehyde-3-Aminopyridine Schiff base (3-APY-{SalH})

The Schiff base was prepared by mixing equimolecular quantity of *o*-hydroxybenzaldehyde (0.08 mole, 9.76 g) and 3-aminopyridine (0.08 mole, 7.52 g) in 10 mL methanol in a round bottomed flask equipped with a condenser⁶⁴. The reaction mixture was refluxed on water bath for 4 hours. After completion of reaction, the reaction mixture was cooled, filtered and dried in a desiccator over anhydrous CaCl₂ and recrystallized from methanol, a sharp yellowish orange crystalline product was obtained.

Preparation of complexes

Tellurium tetrachloride, organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides, when reacted with Schiff base (3-APY-{SalH}) form solid complexes as described below:

[TeCl₃(3-APY-{Sal})], [RTeCl₂(3-APY-{Sal})] and [R₂TeCl(3-APY-{Sal})]

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 (3-APY-{SalH}) 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 P₄O₁₀.

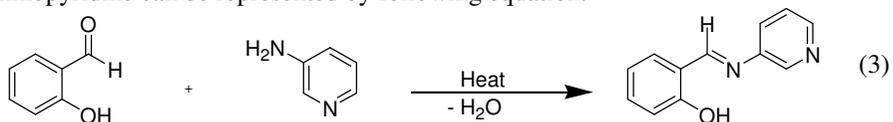
Results and Discussion

TeCl₄ when heated with anisole⁵⁹⁻⁶¹, phenol⁶² and *o*-cresol⁶³ (R-H) appears to undergo the Friedel Craft type condensation reaction where by TeCl₃⁺ unit attacks a position *para* to the

methoxy/hydroxyl group in the aromatic ring, thus resulting in the formation of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides.



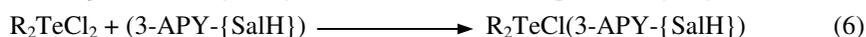
Preparation of Schiff Base (3-APY-{SalH}), by the reaction of *o*-hydroxybenzaldehyde with 3-aminopyridine can be represented by following equation.



o-Hydroxybenzaldehyde 3-aminopyridine

(3-APY-{SalH})

Schiff Base reacts with tellurium(IV) chloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides to yield the coloured tellurium(IV) complexes.



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

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 10.52-36.78 $\text{S cm}^2 \text{mol}^{-1}$ which predict the non-electrolyte to 1:1 weak electrolyte type behavior^{65, 66} of these Schiff base complexes in DMSO, probably due to ionization into $\text{TeCl}_2(3\text{-APY}\{-\text{Sal}\})^+ / \text{RTeCl}(3\text{-APY}\{-\text{Sal}\})^+ / \text{R}_2\text{Te}(3\text{-APY}\{-\text{Sal}\})^+$ 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 3-APY-{Sal}⁻ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes⁶⁷ which are reported to be non-electrolytes.

Infrared spectra

The IR spectral data (Table 2) of Schiff base and its complexes with organyltellurium(IV) chlorides are compared in order to determine the coordination sites involved in the chelation. The vibrational spectra of the ligand exhibit the characteristic bands^{2,37,64,68}, one at 1619 cm^{-1} of azomethine $\nu_{(\text{C}=\text{N})}$ group and another at 3052 cm^{-1} , of phenolic $\nu_{(\text{OH})}$ group. While in the complexes $\nu_{(\text{C}=\text{N})}$ bands were shifted to lower frequency region^{2,13,64,68,69,70} *i.e.* $1605\text{-}1613 \text{ cm}^{-1}$ indicating a decrease in the C=N bond strength due to the formation of coordinate bond of the metal with the imine nitrogen lone pair^{71,72}. The absence of peak due to the phenolic -OH group in free ligand at 3052 cm^{-1} in the complexes suggests the coordination of ligand to the metal through phenolic oxygen via deprotonation which infers that azomethine nitrogen and phenolic oxygen are the coordination sites of bidentate ligand. Also, the appearance of new strong bands around^{69,70,73,74} at $289\text{-}295 \text{ cm}^{-1}$ due to $\nu_{(\text{Te}-\text{O})}$ and in the range of $414\text{-}419 \text{ cm}^{-1}$ due⁷⁵ to $\nu_{(\text{Te}-\text{N})}$ further support the involvement of phenolic oxygen and azomethine nitrogen atom of Schiff base in the coordination.

Table 1. Analytical data, molar conductance and physical properties for Schiff base (3-APY- $\{SalH\}$) complexes of tellurium(IV) values of Λ_M reported^{65,66} for 1:1 electrolytes in DMSO = 50-70 S cm² mol⁻¹

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 ² mol ⁻¹ in DMSO
					C	H	N	Te	Cl	
Schiff Base	(3-APY- $\{SalH\}$)	C ₁₂ H ₁₀ N ₂ O (198.24)	Yellowish Orange (94)	66-68	72.48 (72.70)	5.25 (5.09)	14.02 (14.13)	-	-	-
1	TeCl ₃ (3-APY- $\{Sal\}$)	C ₁₂ H ₉ Cl ₃ N ₂ OTe (431.33)	Dark Orange (71)	182-184	34.72 (34.87)	2.38 (2.11)	6.35 (6.50)	29.42 (29.58)	24.57 (24.69)	11.23
2	RTeCl ₂ (3-APY- $\{Sal\}$) (4-methoxyphenyl)	C ₁₉ H ₁₆ Cl ₂ N ₂ O ₂ Te (502.97)	Reddish Brown (83)	137-139	45.12 (45.37)	3.34 (3.21)	5.35 (5.57)	25.18 (25.37)	13.87 (14.12)	17.11
3	RTeCl ₂ (3-APY- $\{Sal\}$) (4-hydroxyphenyl)	C ₁₈ H ₁₄ Cl ₂ N ₂ O ₂ Te (488.94)	Dark Brown (77)	171-173	44.05 (44.21)	2.97 (2.89)	5.58 (5.73)	26.32 (26.10)	14.38 (14.52)	16.38
4	RTeCl ₂ (3-APY- $\{Sal\}$) (3-methyl-4-hydroxyphenyl)	C ₁₉ H ₁₆ Cl ₂ N ₂ O ₂ Te (502.97)	Dark Yellow (85)	207-209	45.12 (45.37)	3.42 (3.21)	5.35 (5.57)	25.18 (25.37)	13.96 (14.12)	10.52
5	R ₂ TeCl(3-APY- $\{Sal\}$) (4-methoxyphenyl)	C ₂₆ H ₂₃ ClN ₂ O ₃ Te (574.61)	Light Brown (73)	218-220	54.21 (54.34)	4.17 (4.04)	4.71 (4.88)	22.46 (22.21)	6.02 (6.18)	26.19
6	R ₂ TeCl(3-APY- $\{Sal\}$) (4-hydroxyphenyl)	C ₂₄ H ₁₆ ClN ₂ O ₃ Te (546.55)	Light Green (79)	206-208	52.53 (52.74)	3.59 (3.51)	4.98 (5.13)	23.22 (23.35)	6.28 (6.50)	36.78
7	R ₂ TeCl(3-APY- $\{Sal\}$) (3-methyl-4-hydroxyphenyl)	C ₂₆ H ₂₃ ClN ₂ O ₃ Te (574.61)	Light brown (80)	160-162	53.97 (54.34)	3.89 (4.04)	4.63 (4.88)	22.43 (22.21)	6.07 (6.18)	33.36

Further, presence of organyl groups of Rte and R₂Te may result in mixing of certain bands, thus making independent assignments very difficult. Thus, the Schiff base ligand is coordinated to the tellurium atom as uninegative ON bidentate ligand give rise to a six membered chelating ring with tellurium centre.

Table 2. Important IR data (cm⁻¹) of the Schiff Base (3-APY-{SalH}) and complexes

Compd No.	(Phenolic) V _(OH)	(Azomethine) V _(C=N)	V _(Te-O)	V _(Te-N)
(3-APY-{SalH})	3052 s	1619 s	-	-
1	-	1605 s	289 s	419 s
2	-	1608 s	292 s	414 s
3	-	1607 m	290 s	419 s
4	-	1606 sh	294 s	416 s
5	-	1609 s	290 s	419 s
6	-	1613 s	290 s	419 s

(s = strong, m = medium, b = broad, sh = shoulder)

¹H NMR spectra

Further, evidence for the coordination mode of the ligand is obtained by ¹H NMR studies and chemical shift data for the free ligand (3-APY-{SalH}) and its complexes are compiled in Table 3.

Table 3. ¹H NMR spectral data of Schiff base (3-APY-{SalH}) and complexes in DMSO-d₆

Compd No	(Phenolic) -OH δ ppm	(Azomethine) -HC=N δ ppm	(Ar rings protons) δ ppm	-CH ₃ /-OCH ₃ * δ ppm	-OH of Rte/R ₂ Te δ ppm
(3-APY-SalH)}	12.773 (s, 1H)	9.306 (s, 1H)	6.950-8.648 (cm, 8H)	-	-
1	-	10.230 (s, 1H)	6.860-7.945 (cm, 8H)	-	-
2	-	10.273 (s, 1H)	6.849-8.347 (cm, 12H)	3.805 (s, 3H*)	-
3	-	10.274 (s, 1H)	6.855-8.093 (cm, 12H)	-	9.104 (s, 1H)
4	-	10.234 (s, 1H)	7.040-8.347 (cm, 11H)	2.508 (s, 3H)	9.088 (s, 1H)
5	-	10.273 (s, 1H)	6.841-8.231 (cm, 16H)	3.432 (s, 6H*)	-
6	-	10.148 (s, 1H)	7.620-8.729 (cm, 16H)	-	9.098 (s, 2H)

s = singlet, cm = complex multiplet

The ligand is characterized by three signals at 12.773 (singlet), 9.306 (singlet) and 6.950-8.648 (complex multiplet) δ ppm which are assigned to phenolic -OH, azomethine proton -N=CH- and aromatic as well as pyridine protons respectively.

The presence of -N=CH- proton signal at 9.306 δ ppm in the ligand confirms the formation of base by condensation of *o*-hydroxybenzaldehyde with 3-aminopyridine. The azomethine proton signal in the complexes i.e. [TeCl₃(3-APY-{Sal})], [RTeCl₂(3-APY-{Sal})]

and $[R_2TeCl(3-APY-\{Sal\})]$, shows downfield shifting as compared to free ligand, suggesting deshielding of azomethine proton due to coordination to tellurium through the azomethine nitrogen^{48,49,68,76}.

Also the absence of proton signal at 12.773 δ ppm indicating that phenolic proton is absent in complexes and confirms the participation of -OH group of *o*-hydroxybenzaldehyde in the coordination via deprotonation.

Independent assignments to the aryl protons of (3-APY- $\{SalH\}$) and RTe / R_2Te are not possible due to overlapping of signals in this region. On the basis of spectral studies, it may be concluded that (3-APY- $\{SalH\}$) acts as uninegative (*ON*) bidentate ligand, resulting in the formation of pentacoordinated tellurium(IV) complexes and proposed structures are shown in Figure 1.

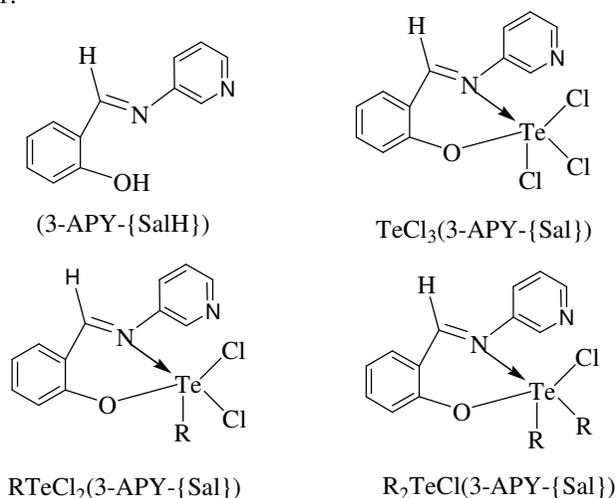


Figure 1. Proposed structures of Schiff base (3-APY- $\{SalH\}$) and tellurium (IV) complexes

Biological studies

The *o*-hydroxybenzaldehyde-3-aminopyridine Schiff base (3-APY- $\{SalH\}$) and newly synthesized organytellurium(IV) Schiff base complexes were evaluated for their antimicrobial activities *in vitro* against Gram-positive bacteria (*Staphylococcus aureus* MTCC 96 and *Streptococcus pyogenes* MTCC 442), Gram-negative bacteria (*Pseudomonas aeruginosa* MTCC 1688 and *Escherichia coli* MTCC 443) and fungi *Candida albicans* MTCC 227, *Aspergillus niger* MTCC 282 and *Aspergillus clavatus* MTCC 1323. The evaluation of the biological activities was carried by “Broth Dilution Method”. The MIC of the control organism is read to check the accuracy of the drug concentrations. The lowest concentration inhibiting growth of the organism is recorded as the MIC. The MIC values of ligand and the complexes have been compared with standard drugs *ampicillin* and *chloramphenicol* for antibacterial, *nystatin* and *greseofulvin* for antifungal activities⁷⁷ are given in Table 4.

The data show the complexes of organytellurium(IV) exhibit more antibacterial activity than antifungal activity as compared to free ligand. The complex no. 3 *i.e.* $[RTeCl_2(3-APY-\{Sal\})]$: where R=4-hydroxyphenyl, shows substantial activity against all four bacterial strain than Schiff base itself. Most of the complexes are moderately more effective against fungal strain *C. albicans* MTCC 227 as compare to free ligand.

Table 4. Minimum inhibitory concentration MIC ($\mu\text{g/mL}$) of Schiff Base (3-APY- $\{\text{SalH}\}$) and complexes

Compd No	Bacterial Strain				Fungal Strain		
	S.	S.	P.	E.	C.	A.	A.
	<i>aureus</i> MTCC 96	<i>pyogenes</i> MTCC 442	<i>aeruginosa</i> MTCC 1688	<i>coli</i> MTCC 443	<i>albicans</i> MTCC 227	<i>niger</i> MTCC 282	<i>clavatus</i> MTCC 1323
(3-APY- $\{\text{SalH}\}$)	500	250	250	250	> 1000	200	500
1	250	250	500	500	> 1000	> 1000	> 1000
2	200	250	250	200	500	1000	1000
3	125	125	100	125	500	1000	1000
6	200	250	250	200	250	500	500
	Standard Drugs						
<i>Ampicillin</i>	250	100	100	100	-	-	-
<i>Chloramphenicol</i>	50	50	50	50	-	-	-
<i>Nystatin</i>	-	-	-	-	100	100	100
<i>Greseofulvin</i>	-	-	-	-	500	100	100

Conclusion

The Schiff base (3-APY- $\{\text{SalH}\}$) has been prepared by condensation of o-hydroxybenzaldehyde with 3-aminopyridine. Schiff base when reacted with tellurium tetrachloride, organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides in 1:1 molar ratios yield $[\text{TeCl}_3(3\text{-APY-}\{\text{Sal}\})]$, $[\text{RTeCl}_2(3\text{-APY-}\{\text{Sal}\})]$ and $[\text{R}_2\text{TeCl}(3\text{-APY-}\{\text{Sal}\})]$; where R=4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl type complexes. Spectral studies predict the pentacoordinated tellurium centre by the monobasic bidentate (ON) Schiff base. Some of these complexes possess substantial antimicrobial activity.

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