

Coordination of Tellurium(IV) with Schiff Base Derived from *o*-Vanillin and 3-Aminopyridine

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Received: November 03, 2017; Accepted: February 24, 2018; Published: February 27, 2018

Abstract

Seven new organytellurium(IV) complexes with monobasic ON bidentate Schiff base ligand have been synthesized. The Schiff base ligand (3-APY-*o*-VanH) was prepared by condensation of *o*-vanillin with 3-aminopyridine. The newly synthesized organytellurium(IV) complexes and Schiff base have been characterized by elemental analyses, conductivity measurements, FT-IR and ¹H NMR spectral studies. The structure of the complexes obtained was confirmed by FT-IR and proton NMR which exhibited pentacoordinated tellurium centre having Ψ-trigonal bipyramidal geometry. Schiff base as well as their organytellurium(IV) complexes were also evaluated for their antimicrobial activities *in vitro* against Gram-positive bacteria (*Staphylococcus aureus* and *Streptococcus pyogenes*), Gram-negative bacteria (*Pseudomonas aeruginosa* and *Escherichia coli*) and fungi *Candida albicans*, *Aspergillus niger* and *Aspergillus clavatus*.

Keywords: *o*-Vanillin, Schiff base ligand, Organytellurium(IV) complexes, Monobasic, Biological activities

Introduction

Schiff base ligands are well-known to be good chelating agents as bidentate, tridentate or polydentate ligands, particularly when the group such as –OH/-SH are present close to azomethine group, resulting in the formation of five or six membered ring complexes [1-6]. Schiff bases are reported to have biological activities like antibacterial [7-15], antifungal [7,9-12,16], antitumor [10,17,18], antiviral [19-21], anti-HIV [22], herbicidal [23] and anti influenza A virus [24] activities.

Tellurium(IV) chloride is also known to form adducts with amides [25,26] and thiourea [27], thus reflecting its acceptor behaviour [1,2,28-33]. Also, organytellurium(IV) chlorides are known [1,2,28-50] 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*-vanillin-3-aminopyridine Schiff base (3-APY-*o*-VanH), to synthesize some new complexes of tellurium(IV).

Citation: Malik A, Goyat G, Vikas K, Verma KK and Garg S. Coordination of Tellurium(IV) with Schiff Base Derived from *o*-Vanillin and 3-Aminopyridine. Int J Chem Sci. 2018;16(1):249

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Materials and Methods

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 [51,52] 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.

4-Methoxyphenyltellurium(IV) trichloride [53,54], bis(p-methoxyphenyl)tellurium(IV) dichloride [54,55], 4-hydroxyphenyltellurium(IV) trichloride [56], bis(p-hydroxyphenyl)tellurium(IV) dichloride [56], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride [57] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride [57] 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 [53-57].

Preparation of o-vanillin-3-aminopyridine Schiff base (3-APY-{o-VanH})

The Schiff base was prepared by mixing equimolecular quantity of o-vanillin (0.08 mole, 12.17 g) and 3-aminopyridine (0.08 mole, 7.52 g) in 25 ml methanol in a round bottomed flask equipped with a condenser [58]. 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, yellowish crystalline product was obtained.

Preparation of complexes

Tellurium tetrachloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides, when reacted with Schiff base (3-APY-{o-VanH}) form solid complexes as described below:

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

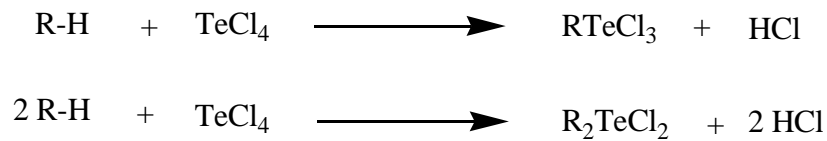
The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL anhydrous methanol to a hot solution of 5 mmol Schiff base (3-APY-{o-VanH}) in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent 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.

Physical studies

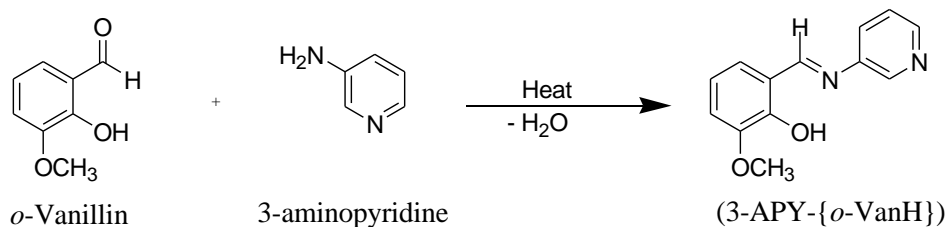
Carbon, hydrogen and nitrogen analyses were obtained microanalytically from SAIF, Panjab University Chandigarh on a Thermo Finnigan CHNS analyser. Conductance studies were performed under dry condition in DMSO at 25 ± 2°C with a dip type conductivity cell on a microprocessor-based conductivity bridge type MICROSIL. Infrared spectra (4000-40 cm⁻¹) were recorded in KBr and Polyethylene pellets for Mid-IR and Far-IR respectively, on a F.T. 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.

Results and Discussion

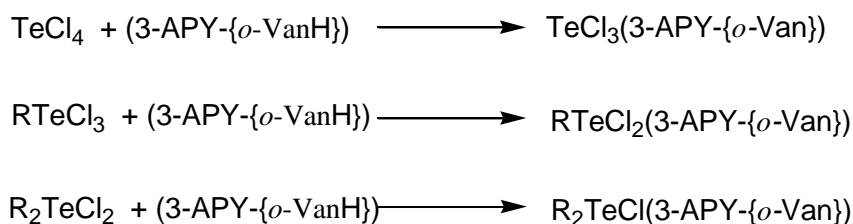
TeCl₄ when heated with anisole [53-55], phenol [56] and o-cresol [57,58] (R-H) appears to undergo the Friedel Craft type condensation reaction where by TeCl₃⁺ unit attacks a position *para* to the methoxy/hydroxyl group in the aromatic ring, thus resulting in the formation of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides.



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



Schiff Base reacts with tellurium(IV) chloride, organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides in 1:1 molar ratio to yield the corresponding organytellurium(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

The molar conductance (Λ_M) data for organytellurium(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 12.01-44.36 S $\text{cm}^2 \text{mol}^{-1}$ which predict the non-electrolyte to 1:1 weak electrolyte type behavior [59,60] of these Schiff base complexes in DMSO, probably due to ionization into $\text{TeCl}_2(3\text{-APY-}\{o\text{-Van}\})^+/\text{RTeCl}(3\text{-APY-}\{o\text{-Van}\})^+/\text{R}_2\text{Te}(3\text{-APY-}\{o\text{-Van}\})^+$ 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-*o*-Van $\}^-$ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes [61] which are reported to be non-electrolytes.

Infrared spectra

The IR spectral data of Schiff base and its complexes with organytellurium(IV) chlorides are recorded in solid state and selected bands of diagnostic importance are collected in TABLE 2. The band at 1616 cm^{-1} of the ligand is assigned [25,58,62] to the stretching vibration of the azomethine group. When the spectra of complexes are compared with those of uncomplexed Schiff base ligand the $\nu_{\text{C=N}}$ band is shifted to lower frequency [16,58,62-64] this indicates that imine nitrogen [65,66] is

coordinated to the metal centre. The band around 3085 cm^{-1} , in free ligand ascribed to the $\nu_{(\text{OH})}$ of phenolic group disappear on complexation with tellurium atom and shows that phenolic group of o-vanillin is involved in bonding after deprotonation. The two new bands appear in range $289\text{-}294\text{ cm}^{-1}$ and $408\text{-}419\text{ cm}^{-1}$ assigned to $\nu_{(\text{Te-O})}$ [63,64,67] and $\nu_{(\text{Te-N})}$ mode of vibration. Thus, IR data predict to monobasic bidentate nature of the Schiff base (3-APY-{o-VanH}) involving azomethine nitrogen atom and phenolic oxygen after deprotonation giving rise to six membered chelate ring with the penta coordinated tellurium centre.

TABLE 1. Analytical data, molar conductance and physical properties for Schiff base (3-APY-{o-VanH}) complexes of tellurium(IV). Values of Λ_M reported [59,60] for 1:1 electrolytes in DMSO= $50\text{-}70\text{ S cm}^2\text{ mol}^{-1}$.

Compound No.	Complex (R)	Empirical Formula (Formula Wt.)	Colour Yield, (%)	M. Pt. ($^{\circ}\text{C}$) dec.	Analyses % Found (Calculated)					Λ_M at ca. $10^{-3}\text{M S cm}^2\text{ mol}^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	(3-APY-{o-VanH})	$\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_2$ (228.15)	Yellowish (90)	118-120	68.24 (68.43)	5.32 (5.26)	12.17 (12.28)	-	-	-
I	$\text{TeCl}_3(3\text{-APY}\text{-}\{\text{o-Van}\})$	$\text{C}_{13}\text{H}_{11}\text{Cl}_3\text{N}_2\text{O}_2\text{Te}$ (461.24)	Dark Brown (85)	204-206	33.71 (33.85)	2.45 (2.38)	6.15 (6.07)	27.53 (27.66)	22.95 (23.09)	21.16
II	$\text{RTeCl}_2(3\text{-APY}\text{-}\{\text{o-Van}\})$ (4-methoxyphenyl)	$\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_3\text{Te}$ (532.81)	Reddish brown (76)	138-140	44.92 (45.08)	3.22 (3.38)	5.18 (5.26)	24.05 (23.95)	13.03 (13.14)	18.09
III	$\text{RTeCl}_2(3\text{-APY}\text{-}\{\text{o-Van}\})$ (4-hydroxyphenyl)	$\text{C}_{19}\text{H}_{16}\text{Cl}_2\text{N}_2\text{O}_3\text{Te}$ (518.80)	Brick Red (69)	153-155	43.77 (43.98)	3.23 (3.08)	5.27 (5.40)	24.67 (24.59)	13.54 (13.69)	12.01
IV	$\text{RTeCl}_2(3\text{-APY}\text{-}\{\text{o-Van}\})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_3\text{Te}$ (532.81)	Brown (74)	190-192	44.89 (45.08)	3.45 (3.38)	5.34 (5.26)	23.86 (23.95)	13.20 (13.14)	19.12
V	$\text{R}_2\text{TeCl}(3\text{-APY}\text{-}\{\text{o-Van}\})$ (4-methoxyphenyl)	$\text{C}_{27}\text{H}_{25}\text{ClN}_2\text{O}_4\text{Te}$ (604.38)	Dark Yellow (79)	184-186	53.52 (53.65)	4.27 (4.14)	4.53 (4.64)	21.23 (21.11)	5.82 (5.87)	32.19
VI	$\text{R}_2\text{TeCl}(3\text{-APY}\text{-}\{\text{o-Van}\})$ (4-hydroxyphenyl)	$\text{C}_{25}\text{H}_{21}\text{ClN}_2\text{O}_4\text{Te}$ (576.36)	Brown (82)	192-194	51.87 (52.09)	3.51 (3.64)	4.74 (4.86)	22.05 (22.14)	6.09 (6.16)	42.90
VII	$\text{R}_2\text{TeCl}(3\text{-APY}\text{-}\{\text{o-Van}\})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{27}\text{H}_{25}\text{ClN}_2\text{O}_4\text{Te}$ (604.38)	Reddish Brown (78)	144-146	53.45 (53.65)	4.03 (4.14)	4.56 (4.64)	21.02 (21.11)	5.75 (5.87)	44.36

^1H NMR spectra

The ^1H NMR spectral data of ligand (3-APY-{o-VanH}) and complexes were recorded in DMSO- d_6 and are given in TABLE 3. The proton peak of phenolic $-\text{OH}$ group at $13.124\text{ }\delta$ ppm had disappeared, which suggest that the hydroxyl group coordinates to the metal centre after deprotonation. The singlet at $8.660\text{ }\delta$ ppm (s, 1H) attributed to the imine hydrogen in the ligand shift to downfield side in complexes clearly demonstrate [42,43,62] the coordination of azomethine nitrogen to tellurium. Independent assignments to the aryl protons of (3-APY-{o-VanH}) and $\text{RTe}/\text{R}_2\text{Te}$ are not possible due to overlapping of signals in this region.

TABLE 2. Important IR data (cm⁻¹) of the Schiff base (3-APY-{*o*-VanH}) and Complexes.

Compound No.	(Phenolic) ν _(OH)	(Azomethine) ν _(C=N)	ν _(Te-O)	ν _(Te-N)
(3-APY-{ <i>o</i> -VanH})	3085 s	1616 s	-	-
I	-	1606 s	289 m	419 m
II	-	1609 s	290 s	419 s
III	-	1609 m	289 m	418 s
IV	-	1608 sh	290 s	419 m
V	-	1606 s	294 s	408 mb
VI	-	1609 s	291 m	419 m
VII	-	1609 s	292 s	414 s
s=strong, m=medium, b=broad, sh=shoulder				

TABLE 3. ¹H NMR spectral data of Schiff base (3-APY-{*o*-VanH}) and complexes in DMSO-d₆. s=singlet, cm=complex multiplet. Spectra of compound number VI not well resolved due to poor solubility.

Compound Number	(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-{ <i>o</i> -VanH})	13.124 (s, 1H)	8.660 (s, 1H)	6.899-8.582 (cm, 7H)	3.954 (s, 3H*)	-
I	-	10.140 (s, 1H)	6.841-7.944 (cm, 7H)	3.749 (s, 3H*)	-
II	-	10.140 (s, 1H)	6.832-8.230 (cm, 11H)	3.414 (s, 6H*)	-
III	-	10.138 (s, 1H)	6.838-8.229 (cm, 11H)	3.424 (s, 3H*)	9.089 (s, 1H)
IV	-	10.273 (s, 1H)	6.851-8.093 (cm, 10H)	2.513 (s, 3H)/ 3.848 (s, 3H*)	9.102 (s, 1H)
V	-	10.147 (s, 1H)	6.811-7.929 (cm, 15H)	3.402 (s, 9H*)	-
VII	-	10.145 (s, 1H)	6.850-8.095 (cm, 13H)	2.510 (s, 6H)/ 3.437 (s, 3H*)	9.098 (s, 2H)
s =singlet, cm=complex multiplet. Spectra of compound number VI not well resolved due to poor solubility.					

Conclusion

The Schiff base (3-APY-{*o*-VanH}) and newly synthesized organytellurium(IV) Schiff base complexes were screened *in vitro* antimicrobial potential against Gram +ve bacteria (*S. aureus* MTCC 96 and *S. pyogenes* MTCC 442), Gram -ve bacteria (*P. aeruginosa* MTCC 1688 and *E. coli* MTCC 443) strain; fungal strains *C. albicans* MTCC 227, *A. niger* MTCC 282 and *A. clavatus* MTCC 1323 by “Broth Dilution Method”. The results were recorded in terms of MIC values are present in TABLE 4. Comparative study of MIC value for Schiff base (3-APY-{*o*-VanH}) and their tellurium(IV) complexes indicates that some complexes exhibit higher antibacterial activity than Schiff base itself. It has been observed that the complex no. VI [R₂TeCl(3-APY-{*o*-Van})]: where R=4-hydroxyphenyl,) shows stronger antifungal activity than Schiff base itself against *A. niger* and *A. Clavatus*.

On the basis of these studies, the proposed structures for the complexes are as below (FIG. 1).

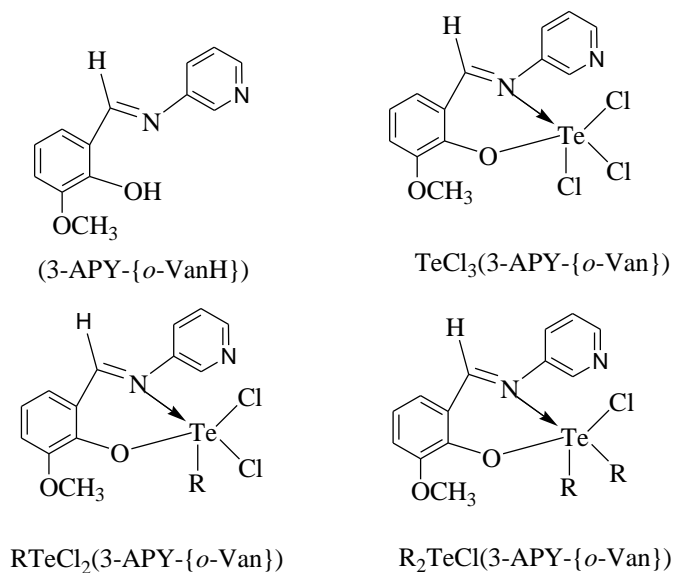


FIG. 1. Proposed structures of Schiff base (3-APY-{*o*-VanH}) and tellurium(IV) complexes.

TABLE 4. Minimum inhibitory concentration MIC ($\mu\text{g/mL}$) of Schiff base (3-APY-{*o*-VanH}) and complexes.

Compound Number	Bacterial Strain				Fungal Strain		
	<i>S. aureus</i> MTCC 96	<i>S. pyogenes</i> MTCC 442	<i>P. aeruginosa</i> MTCC 1688	<i>E. coli</i> MTCC 443	<i>C. albicans</i> MTCC 227	<i>A. niger</i> MTCC 282	<i>A. clavatus</i> MTCC 1323
(3-APY-{ <i>o</i> -VanH})	250	250	200	200	250	> 1000	> 1000
I	250	250	125	100	> 1000	> 1000	> 1000
II	250	200	100	125	500	1000	1000
III	200	200	250	250	500	1000	1000
VI	500	500	250	250	1000	250	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

Acknowledgement

The authors are grateful to M. D. University, Rohtak for providing the necessary facilities. One of the authors (AM) is also thankful to HSCST, Haryana for providing a fellowship. We also thank Microcare Laboratory, Surat, for providing antimicrobial activities.

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