

Synthesis, Spectral and Antimicrobial Studies of Some *O*-Vanillin-2-aminopyridine Schiff Base Complexes of Organyltellurium(IV)

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Abstract: The reactions of tellurium(IV) derivatives, TeCl_4 , RTeCl_3 and R_2TeCl_2 with monobasic *ONN* tridentate Schiff base ligand derived by the condensation of *o*-vanillin with 2-aminopyridine have been carried out to give tellurium(IV) complexes, $[\text{TeCl}_3(2\text{-APY-}\{o\text{-Van}\})]$, $[\text{RTeCl}_2(2\text{-APY-}\{o\text{-Van}\})]$ and $[\text{R}_2\text{TeCl}(2\text{-APY-}\{o\text{-Van}\})]$: where R = *p*-methoxyphenyl, *p*-hydroxyphenyl, 3-methyl-4-hydroxyphenyl. The synthesized Schiff Base (2-APY-*o*-VanH) and their complexes were characterized by elemental analyses, conductivity measurements, FT-IR and proton NMR spectral studies. The Schiff base and some of their tellurium(IV) complexes display antifungal and antibacterial activities.

Keywords: Tridentate Schiff base, *o*-Vanillin, Organyltellurium, Antifungal, Antibacterial activities

Introduction

Schiff base derived from aromatic aldehydes have arisen the researchers interest because of its varied use in biological systems as antitubercular agent^{1,2}, antibacterial^{3,4,9}, antifungal⁶⁻¹⁰ and antitumor activities^{11,12}. Schiff bases are known to be medicinally important and are used to design medicinal compounds¹³. Some Schiff bases bearing aryl groups or heterocyclic residues possess excellent biological activities have attracted the attention of many researchers in recent years^{14,15}. The Schiff bases derived from *o*-vanillin and 2,3-diaminopyridine have been used as ionophores in a Cu(II) selective electrochemical sensors¹⁶.

Organyltellurium(IV) chlorides are known¹⁷⁻⁴¹ 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 organyltellurium(IV) chlorides with *o*-vanillin-2-aminopyridine Schiff base 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 N_2 atmosphere. The solvents were purified by standard method^{42,43}

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 organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides

4-Methoxyphenyltellurium(IV) trichloride^{44,45}, bis(*p*-methoxyphenyl)tellurium(IV)dichloride^{45,46}, 4-hydroxyphenyltellurium(IV)trichloride⁴⁷, bis(*p*-hydroxyphenyl)tellurium(IV)dichloride⁴⁷, 3-methyl-4-hydroxyphenyltellurium(IV)trichloride⁴⁸ and bis(3-methyl-4-hydroxyphenyl)-tellurium(IV)dichloride⁴⁸ 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 literature⁴⁴⁻⁴⁸.

*Preparation of Schiff base (2-APY-{*o*-VanH})*

The Schiff base was prepared by mixing equimolecular quantity of *o*-vanillin (0.08 mole, 12.17 g) and 2-aminopyridine (0.08 mole, 7.52 g) in 25 mL methanol in a round bottom flask equipped with a condenser⁴⁹. 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 dark orange crystalline product was dried in a desiccator over anhydrous CaCl₂ and kept over P₄O₁₀. Yield = 93%, m.pt. (decomp.) = 82-84 °C.

Preparation of complexes

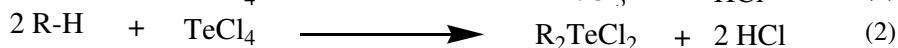
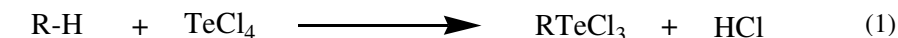
Tellurium tetrachloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides, when reacted with Schiff base (2-APY-{*o*-VanH}) form solid complexes as described below.

*[TeCl₃(2-APY-{*o*-Van})], [RTeCl₂(2-APY-{*o*-Van})] and [R₂TeCl(2-APY-{*o*-Van})]*

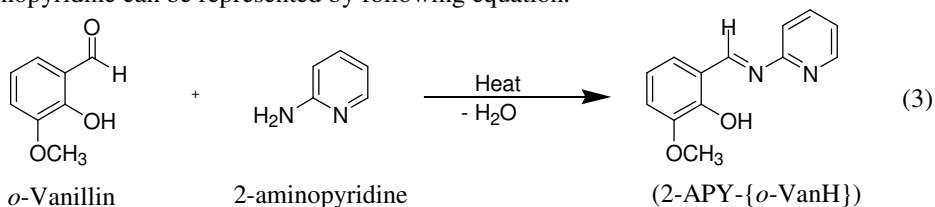
The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL anhydrous methanol to a hot solution of 5 mmol Schiff base (2-APY-{*o*-VanH}) in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent 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.

Results and Discussion

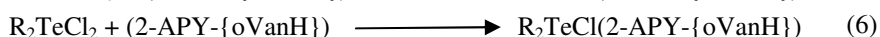
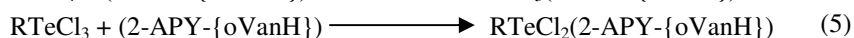
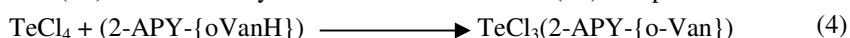
Anisole⁴⁴⁻⁴⁶, phenol⁴⁷ and *o*-cresol⁴⁸ (R-H) undergo Friedel Craft type condensation reaction when reacted with tellurium tetrachloride in boiling organic solvents to form organyltellurium(IV)trichlorides and diorganyltellurium(IV)dichlorides. This reaction involves the electrophilic substitution of the aromatic ring by a trichlorotellurium group at a position *para* to the methoxy / hydroxyl groups.



Preparation of Schiff base (2-APY-*o*-VanH)), by the reaction of *o*-vanillin with 2-aminopyridine can be represented by following equation.



Schiff base reacts with tellurium(IV)chloride, organytellurium(IV)trichlorides and diorganytellurium(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 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 8.53-40.56 $\text{S cm}^2 \text{mol}^{-1}$ which predict the non-electrolyte to 1:1 weak electrolyte type behavior^{50,51} of these Schiff base complexes in DMSO, probably due to ionization into $\text{TeCl}_2(2\text{-APY-}\{o\text{-Van}\})^+ / \text{RTeCl}(2\text{-APY-}\{o\text{-Van}\})^+ / \text{R}_2\text{Te}(2\text{-APY-}\{o\text{-Van}\})^+$ 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 $2\text{-APY-}\{o\text{-Van}\}^-$ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes⁵² which are reported to be non-electrolytes.

Infrared spectra

Characteristics FT-IR bands of the free ligand and its complexes with their assignment are presented in Table 2. The FT-IR spectrum of the ligand^{10,49,53-55} showed a $\nu_{(\text{C}=\text{N})}$ peak at 1610 cm^{-1} , $\nu_{(\text{C}-\text{N}-\text{C})}$ peak at 1462 cm^{-1} and the absence of $\nu_{(\text{C}=\text{O})}$ at 1643 cm^{-1} and $\nu_{(\text{NH}_2)}$ peaks around $3250\text{-}3300 \text{ cm}^{-1}$ is because of Schiff base condensation.

The spectrum also showed a broad band at $\approx 3064 \text{ cm}^{-1}$ due to $\nu_{(\text{OH})}$. The strong band at 1610 cm^{-1} in the IR spectrum of free ligand assigned to $\nu_{(\text{C}=\text{N})}$ that changes in the FT-IR spectra of the complexes^{6,49,53-55} (towards lower frequency region), indicating coordination through azomethine nitrogen^{56,57}.

The $\nu_{(\text{C}-\text{N}-\text{C})}$ strong band at 1462 cm^{-1} in the free ligand shifts to higher frequency upon complexation due to coordination with nitrogen atom of pyridine ring. The broad band appearing around 3064 cm^{-1} in free ligand assignable to $\nu_{(\text{OH})}$ vibration mode is absent in the spectra of complexes suggesting the coordination through oxygen of phenolic group to the tellurium centre.

Table 1. Analytical data, molar conductance and physical properties for Schiff base (2-APY-{*o*-VanH}) complexes of tellurium(IV) values of Λ_M reported^{50,51} 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	(2-APY-{ <i>o</i> -VanH})	C ₁₃ H ₁₂ N ₂ O ₂ (228.15)	Dark orange (93)	82-84	68.28 (68.43)	5.43 (5.26)	12.14 (12.28)	-	-	-
1	TeCl ₃ (2-APY-{ <i>o</i> -Van})	C ₁₃ H ₁₁ Cl ₃ N ₂ O ₂ Te (461.24)	Light brown (78)	193-195	33.61 (33.85)	2.47 (2.38)	5.96 (6.07)	27.54 (27.66)	22.95 (23.09)	8.53
2	RTeCl ₂ (2-APY-{ <i>o</i> -Van}) (4-methoxyphenyl)	C ₂₀ H ₁₈ Cl ₂ N ₂ O ₃ Te (532.81)	Yellowish green (76)	174-176	44.89 (45.08)	3.52 (3.38)	5.15 (5.26)	23.84 (23.95)	13.02 (13.14)	15.92
3	RTeCl ₂ (2-APY-{ <i>o</i> -Van}) (4-hydroxyphenyl)	C ₁₉ H ₁₆ Cl ₂ N ₂ O ₃ Te (518.80)	Dark brown (82)	203-205	43.75 (43.98)	3.20 (3.08)	5.23 (5.40)	24.47 (24.59)	13.52 (13.69)	19.08
4	RTeCl ₂ (2-APY-{ <i>o</i> -Van}) (3-methyl-4-hydroxyphenyl)	C ₂₀ H ₁₈ Cl ₂ N ₂ O ₃ Te (532.81)	Brick red (77)	181-183	44.92 (45.08)	3.24 (3.38)	5.18 (5.26)	23.82 (23.95)	13.04 (13.14)	16.33
5	R ₂ TeCl(2-APY-{ <i>o</i> -Van}) (4-methoxyphenyl)	C ₂₇ H ₂₅ ClN ₂ O ₄ Te (604.38)	Light brown (75)	197-199	53.52 (53.65)	4.03 (4.14)	4.52 (4.64)	21.02 (21.11)	5.75 (5.87)	24.67
6	R ₂ TeCl(2-APY-{ <i>o</i> -Van}) (4-hydroxyphenyl)	C ₂₅ H ₂₁ ClN ₂ O ₄ Te (576.36)	Yellowish brown (85)	200-202	51.94 (52.09)	3.74 (3.64)	4.72 (4.86)	22.04 (22.14)	6.05 (6.16)	40.56
7	R ₂ TeCl(2-APY-{ <i>o</i> -Van}) (3-methyl-4-hydroxyphenyl)	C ₂₇ H ₂₅ ClN ₂ O ₄ Te (604.38)	Dark brown (73)	177-179	53.47 (53.65)	4.25 (4.14)	4.52 (4.64)	20.97 (21.11)	5.73 (5.87)	39.93

In the spectra of complexes, the bands observed in the 289- 290 cm^{-1} and 418-419 cm^{-1} region might be due to $\nu_{(\text{Te-O})}$ ^{54,55,58-60} and $\nu_{(\text{Te-N})}$ ⁶⁰ further support the involvement of phenolic oxygen, azomethine and pyridine nitrogen atoms of Schiff base in the coordination. Further, presence of aryl 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 ONN tridentate ligand give rise to two chelate (six and four membered) with ring tellurium centre.

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

Compound No.	(Phenolic) $\nu_{(\text{OH})}$	(Azomethine) $\nu_{(\text{C=N})}$	(Pyridine) $\nu_{(\text{C-N-C})}$	$\nu_{(\text{Te-O})}$	$\nu_{(\text{Te-N})}$
(2-APY-{ <i>o</i> -VanH})	3064 b	1610 s	1462 s	-	-
1	-	1586 sh	1488 s	289 s	419 s
2	-	1577 s	1489 s	289 s	419 s
3	-	1596 mb	1487 mb	289 s	418 s
4	-	1576 s	1488 m	289 s	419 s
5	-	1590 s	1489 mb	289 s	419 s
6	-	1592 s	1487 m	290 s	419 s
7	-	1588 s	1489 s	290 s	419 s

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

¹H NMR spectra

DMSO-d₆ was used as a solvent to measure the ¹H NMR spectra of free ligand as well as its complexes and data is presented in Table 3.

Table 3. ¹H NMR spectral data of Schiff base (2-APY-{*o*-VanH}) and complexes

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
(2-APY-{ <i>o</i> -VanH})	13.935 (s, 1H)	9.446 (s, 1H)	6.864-8.519 (cm, 7H)	3.944 (s, 3H*)	-
1	-	10.223 (s, 1H)	6.859-7.941 (cm, 7H)	3.996 (s, 3H*)	-
2	-	10.234 (s, 1H)	6.848-8.330 (cm, 11H)	3.810 (s, 6H*)	-
3	-	10.234 (s, 1H)	6.844-8.101 (cm, 11H)	3.420 (s, 3H*)	10.020 (s, 1H)
4	-	10.270 (s, 1H)	6.842-8.240 (cm, 10H)	2.509 (s, 3H)/ 3.841 (s, 3H*)	9.101 (s, 1H)
5	-	10.234 (s, 1H)	6.849-7.932 (cm, 15H)	3.372 (s, 9H*)	-
7	-	10.276 (s, 1H)	6.832-8.099 (cm, 13H)	2.508 (s, 6H)/ 3.388 (s, 3H*)	10.009 (s, 2H)

s = singlet, *cm* = complex multiplet, Spectra of compound number 6 not well resolved due to poor solubility

The ^1H NMR spectrum of the ligand showed signal at δ 9.446 ppm corresponding to azomethine proton. The singlet observed at about δ 13.935 ppm due to phenolic proton of the ligand. The complex multiplets corresponding aromatic protons appeared between δ 6.864-8.519 ppm. The hydroxyl proton (phenolic -OH) disappears in the tellurium(IV) complexes indicating that the -OH group has been deprotonated and bonded to metal centre.

Another signal displays a downfield shift from 9.446 to 10.223-10.276 δ ppm, suggest the decrease of electron density and deshielding of azomethine proton, as a result of which nitrogen atom of azomethine group coordination to the tellurium centre^{30,31,49,53,61}. Independent assignments to the aryl protons of Schiff base and $\text{RTe}/\text{R}_2\text{Te}$ are not possible due to overlapping of signals in this region.

On the basis of spectral studies, it may be concluded that Schiff base acts as uninegative (*ONN*) tridentate ligand, resulting in the formation of hexacoordinated tellurium(IV) complexes probably in a distorted octahedral geometry and proposed structures are shown in Figure 1.

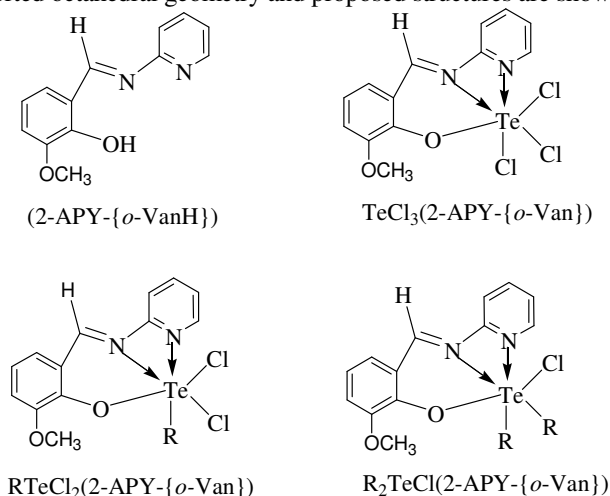


Figure 1. Proposed structures of Schiff base (2-APY-*{o-VanH}*) and tellurium(IV) complexes

Biological studies

The *o*-vanillin-2-aminopyridine Schiff base (2-APY-*{o-VanH}*) and newly synthesized organytellurium(IV) Schiff base complexes were evaluated for their antimicrobial activities *in vitro* against Gram-positive bacteria (*S. aureus* MTCC 96 and *S. pyogenes* MTCC 442), Gram-negative bacteria (*P. aeruginosa* MTCC 1688 and *E. coli* MTCC 443) and fungi *C. albicans* MTCC 227, *A. niger* MTCC 282 and *A. clavatus* MTCC 1323. The evaluation of the biological activities was carried by “Broth Dilution Method”. After incubation the inhibition was measured and the activity of complexes was compared with standard drugs ampicillin and chloramphenicol for antibacterial, nystatin and greseofulvin for antifungal activities⁶². The data is given in Table 4.

The data show the complexes of organytellurium(IV) exhibited more antibacterial activity than antifungal activity as compared to free ligand. The complex no. 3 *i.e.* [$\text{RTeCl}_2(2\text{-APY-}\{o\text{-Van}\})$]: where R = 4-hydroxyphenyl, show substantial activity against two bacterial strains (*S. aureus* and *S. pyogenes*) than Schiff base itself as well as other complexes and even comparable to standard drug ampicillin and complex no. 1 *i.e.* [TeCl_3

(2-APY-*o*-Van)) showed good antibacterial activity against two bacterial strains (*P. aeruginosa* and *E. coli*). Complex no. 2 and 6 *i.e.* [R₂TeCl₂(2-APY-*o*-Van)]: where R = 4-methoxyphenyl and [R₂TeCl₂(2-APY-*o*-Van)]: where R = 4-hydroxyphenyl respectively, showed moderate antibacterial activity against all four pathogenic bacterial strains.

Table 4. Minimum inhibitory concentration MIC (µg/mL) of Schiff base (2-APY-*o*-VanH) and complexes

Compound No.	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
(2-APY- <i>o</i> -VanH)	500	500	250	250	500	1000	1000
1	500	500	100	62.5	1000	500	1000
2	250	250	200	200	1000	500	1000
3	100	100	500	500	500	1000	> 1000
6	250	250	200	250	1000	> 1000	> 1000
Standard Drugs							
Ampicillin	250	100	100	100	-	-	-
Chloramphenicol	50	50	50	50	-	-	-
Nystatin	-	-	-	-	100	100	100
Greseofulvin	-	-	-	-	500	100	100

Conclusion

Schiff base (2-APY-*o*-VanH) derived from *o*-vanillin with 2-aminopyridine have been synthesized. Schiff base when reacted with tellurium tetrachloride, organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides in 1:1 molar ratios yield [TeCl₃(2-APY-*o*-Van)], [R₂TeCl₂(2-APY-*o*-Van)] and [R₂TeCl₂(2-APY-*o*-Van)]: where R=4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl type complexes. The structure of the purified products was confirmed by FT-IR, ¹H NMR spectral studies which predict the hexacoordinated tellurium(IV) complexes probably in a distorted octahedral geometry by the monobasic tridentate (*ONN*) Schiff base. Some of these complexes possess good antimicrobial activity.

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