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RESEARCH ARTICLE

Investigation on Some Tellurium(IV) Complexes of Bidentate(*ON*) Schiff Base Derived from *o*-Vanillin and 2-Aminophenol

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Abstract: Eight new organyltellurium(IV) chelates with bidentate Schiff base formed by condensation of *o*-vanillin and 2-aminophenol having formula *o*-VAPH.RTeCl$_2$, *o*-VAPH.R$_2$TeCl have been synthesized and characterized by elemental analyses, molar conductance, infrared and proton nuclear magnetic resonance spectral studies. The data predict the pentacoordination of tellurium atom by monobasic bidentate(*ON*) Schiff base having Ψ-trigonal bipyramidal geometry. Some of these complexes have also been observed to possess antifungal and antibacterial activity.

Keywords: *o*-Vanillin, 2-Aminophenol, Schiff base, Organyltellurium antifungal, Antibacterial activity

Introduction

Schiff base named after Hugo Schiff described[1] the condensation between aldehydes and primary amines. They contain azomethine[2] (C=N) structural unit which forms strong chelate complexes due to excellent donor ability[3-7] contributed by three factors, electron delocalization with extended conjugation[8] electron donor/acceptor side group on different backbone ring and intramolecular hydrogen bonding[9]. They have various applications such as coordinating ligands[10-15], as catalysts[16-18], in electrochemistry[19-20] and medicinal values. Schiff base and their complexes possess antibacterial[21-26], antifungal[27-32], antiviral[33-36], anticancer[2,36-40] and other biological properties[8,41-45] due to the synergetic effect[46] of reversible binding of oxygen[47], azomethine linkage[48] and hydrogen bonding between OH hydrogen and C=N nitrogen atom[49,50].

The present study has thrown more light on the chelating behaviour of Schiff base derived from *o*-vanillin and 2-aminophenol towards organyltellurium(IV) chlorides which are known[51-64] to act as Lewis acid. These complexes have been examined for their antimicrobial activity against different strains of bacteria and fungi.

Experimental

All preparations were carried out under dry N_2 atmosphere and the solvents used were purified by standard method[65,66] 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 on a Thermo-Finnigan CHNS analyser from SAIF, Panjab University Chandigarh. Conductivity was measured in DMSO at 25±2 ^oC with a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra were recorded in KBr pellets on a FT-Infrared spectrophotometer model RZX (Perkin Elmer) at SAIF, Panjab University Chandigarh. Proton magnetic resonance spectra were recorded in DMSO-d_6 using TMS as an internal reference on BRUKER AVANCE II 400 NMR spectrometer. The antimicrobial screening was carried out by tube dilution method at Department of Pharmaceutical Sciences, M. D. University, Rohtak.

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

4-Methoxyphenyltellurium(IV) trichloride[67,68], bis(4-methoxy-phenyl)tellurium(IV) dichloride[68,69], 4-ethoxyphenyltellurium(IV) trichloride[70], bis(4-ethoxyphenyl)tellurium dichloride[70] 4-hydroxy-phenyltellurium(IV) trichloride[71], bis(4-hydroxyphenyl)tellurium(IV) dichloride[71], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride[72] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride[72] were prepared by the reactions of $TeCl_4$ with anisole/phenetole/phenol/*o*-cresol by the methods as reported in the literature[67-72].

Preparation of o-Vanillin-aminophenol Schiff base ligand (o-VAPH$_2$)[73]

The Schiff base ligand was prepared by condensation of saturated methanolic solutions of *o*-vanillin (0.03 g, 10 mmol) and 2-aminophenol (0.022 g, 10 mmol). The reaction mixture was then refluxed for 3 hours. After cooling, the precipitated Schiff base was collected by filtration and recrystallized from methanol. The orange crystalline product was dried under vacuum or reduced pressure under anhydrous $CaCl_2$ and kept in desiccator over P_4O_{10}. Yield = 75%, m.pt.(decomp.) = 190-192 ^oC (dec.).

Preparation of Schiff base complexes of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides

Organyltellurium(IV) trichlorides, $RTeCl_3$ and diorganyltellurium(IV) dichlorides R_2TeCl_2 (R=4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl), when reacted with sodium salt of Schiff base in equimolar ratio, yield *o*-VAPH.$RTeCl_2$ and *o*-VAPH.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 organyltellurium(IV) trichloride or diorganyltellurium(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 hours, 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

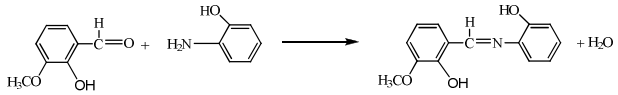
$TeCl_4$ when heated with anisole[67-69], phenetole[70], phenol[71], *o*-cresol[72] (R-H) appears to undergo Friedel-Crafts type condensation reaction whereby $TeCl_3^+$ unit attacks a position

para to the methoxy, ethoxy/hydroxy groups in the aromatic rings, thus resulting in the formation of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides.

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

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

Preparation of Schiff base (*o*-$VAPH_2$) by the reaction of *o*-vanillin and 2-aminophenol can be represented by following equations.



Sodium salt of Schiff base (*o*-$VAPH_2$) *i.e*. o-VAPHNa reacts with organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides in 1:1 molar ratio to yield the corresponding organyltellurium(IV) complexes.

$$o\text{-}VAPH_2 + RTeCl_3 \xrightarrow[-NaCl]{Na/CH_3OH} (o\text{-}VAPH).RTeCl_2 \quad (3)$$

$$o\text{-}VAPH_2 + R_2TeCl_2 \xrightarrow[-NaCl]{Na/CH_3OH} (o\text{-}VAPH).R_2TeCl \quad (4)$$

All the tellurium(IV) complexes are coloured, crystalline solids, stable at room temperature and non-hygroscopic in nature. The complexes have been analysed for their tellurium, chlorine, carbon, hydrogen and nitrogen contents and the data along with their physical properties and yields are presented in Table 1.

Conductance studies

Molar conductance (Λ_M) data for the complexes in DMSO are complied in Table 1. Molar conductance, Λ_M data at *ca.* 10^{-3} M for organyltellurium(IV) complexes in DMSO lie in the range 24.51-42.71 S cm^2 mol^{-1} which predict them as weak electrolyte[74,75] in DMSO, probably due to ionization into RTeCl. *o*-$VAPH^+$/ R_2Te.*o*-$VAPH^+$ and Cl^-. This conductance behavoiur of tellurium (IV) Schiff base complexes is different from those of transition metal complexes[76], which are reported to be non-electrolytes.

Infrared spectra

The IR spectra of Schiff base and its complexes with organyltellurium(IV) are compared in order to determine the coordination sites involved in the chelation. The position or intensities of some guide peaks in the spectrum of ligand are changed upon chelation. The characteristic peaks are listed in Table 2.

Upon comparison it is found that:

1. Examination of Schiff base spectrum shows the presence of a weak band at 2616 cm^{-1} due to intramolecular hydrogen bonding between hydrogen atom of hydroxyl group present on *o*-vanillinidene part and lone pair on nitrogen atom of azomethine group by forming six membered conjugate chelate ring[77-79]. This band disappears on the complexation, which indicates that this hydroxyl group coordinates to tellurium after deprotonation[80-82].
2. A broad band at 3424 cm^{-1} assigned to second hydroxyl group present on aminophenol part which form weak hydrogen bonding to the pi-electron of the azomethine(C=N bond) group[77-79]. This band is still broad in all complexes which render it difficult to attribute to the involvement of this phenolic –OH group in coordination[81-83]. These two hydrogen bonding shown in Figure 1.

Table 1. Analytical data, molar conductance and physical properties of Schiff base and complexes

Compound	Complex (R)	Empirical formula (Formula Wt.)	M.Pt, °C dec.	Colour, Yield, %	Analyses % Found (Calculated)					Λ_M at *ca.* 10^{-3} M S cm^2mol^{-1} in DMSO
					C	H	N	Te	Cl	
Schiff Base	*o*-$VAPH_2$	$C_{14}H_{13}NO_3$ (243.26)	190-192	Orange (75)	68.62 (69.12)	4.88 (5.39)	5.50 (5.76)	-	-	-
1	(*o*-VAPH).$RTeCl_2$ (4-Methoxyphenyl)	$C_{21}H_{19}Cl_2NO_4Te$ (547.89)	128-130	Light brown (90)	45.88 (46.04)	3.38 (3.50)	2.44 (2.56)	23.05 (23.29)	12.74 (12.94)	32.74
2	(*o*-VAPH).$RTeCl_2$ (4-Ethoxyphenyl)	$C_{22}H_{21}Cl_2NO_4Te$ (561.91)	142-144	Brown (84)	46.78 (47.02)	3.65 (3.77)	2.25 (2.49)	22.50 (22.71)	12.51 (12.62)	42.11
3	(*o*-VAPH).$RTeCl_2$ (4-Hydroxyphenyl)	$C_{20}H_{17}Cl_2NO_4Te$ (533.86)	118-120	Dark green (88)	44.85 (45.00)	3.50 (3.21)	2.45 (2.62)	23.85 (23.90)	13.15 (13.28)	35.15
4	(*o*-VAPH).$RTeCl_2$ (3-Methyl-4-hydroxyphenyl)	$C_{21}H_{19}Cl_2NO_4Te$ (547.89)	102-104	Dark brown (82)	45.95 (46.04)	3.35 (3.50)	2.30 (2.56)	23.10 (23.29)	12.77 (12.94)	32.34
5	(*o*-VAPH).R_2TeCl (4-Methoxyphenyl)	$C_{28}H_{26}ClNO_5Te$ (619.56)	98-100	Green (86)	53.84 (54.28)	4.41 (4.23)	2.08 (2.26)	20.80 (20.60)	5.55 (5.72)	33.86
6	(*o*-VAPH).R_2TeCl (4-Ethoxyphenyl)	$C_{30}H_{30}ClNO_5Te$ (647.62)	136-138	Brown (78)	55.45 (55.64)	4.53 (4.67)	2.04 (2.16)	19.55 (19.70)	5.35 (5.47)	40.57
7	(*o*-VAPH).R_2TeCl (4-Hydroxyphenyl)	$C_{26}H_{22}ClNO_5Te$ (591.51)	120-122	Light brown (75)	52.60 (52.79)	3.50 (3.75)	2.15 (2.37)	21.40 (21.57)	5.79 (5.99)	24.51
8	(*o*-VAPH).R_2TeCl (3-Methyl-4-hydroxyphenyl)	$C_{28}H_{26}ClNO_5Te$ (619.56)	188-190	Red (70)	53.98 (54.28)	4.40 (4.23)	2.03 (2.26)	20.85 (20.60)	5.57 (5.72)	42.71

Values of Λ_M reported[74,75] for 1:1 electrolytes in DMSO = 50-70 S cm^2 mol^{-1}

Table 2. Important infrared absorption bands (cm^{-1}) of Schiff Base and complexes

Compound	OH group in 2-aminophenol moiety		OH in *o*-Vanillin		$\nu_{(C=N)}$	$\nu_{(Te-N)}$	$\nu_{(Te-O)}$
	$\nu_{(O-H)}$	$\nu_{(C-O)}$	$\nu_{(O-H)}$	$\nu_{(C-O)}$			
o-$VAPH_2$	3424 mb	1233 s	2614 w	1285 s	1629 s	-	-
1	3442 mb	1234 s	-	1355 s	1632 sh	427 m	290 w
2	3445 mb	1247 s	-	1358 s	1631 sh	424 m	293 w
3	3421 mb	1232 s	-	1357 s	1627 s	427 m	288 w
4	3435 mb	1246 s	-	1354 s	1623 mb	434 s	291 w
5	3458 mb	1255 s	-	1353 s	1639 mb	430 s	294 w
6	3415 mb	1226 s	-	1358 s	1626 s	420 m	298 w
7	3472 mb	1238 s	-	1350 s	1634 mb	430 w	288 w
8	3420 mb	1230 s	-	1360 s	1622 s	432 w	280 w

s=sharp, m=medium, mb=medium broad, sh=shoulder, w=weak

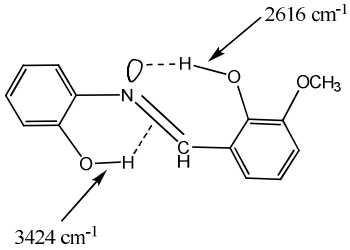


Figure 1. Hydrogen bonding

This makes molecule planar[84-86] in which two OH groups are anti to each other. In planar molecule, the basicity of the azomethine nitrogen atom is higher because its lone pair does not overlap with the aniline ring[87] and also planar molecular has easily available site for coordination[85,88].

3. The involvement of deprotonated -OH group of *o*-vanillinidene part in chelation is confirmed by the blue shift of the phenolic C–O stretching band, observed at 1285 cm^{-1} in the free ligand, to the extent of 40-100 cm^{-1} in the complexes[89]. The band at 1233 cm^{-1} assigned to second hydroxyl group of aminophenol part in Schiff base, does not shift to lower and higher wavenumbers suggesting that this phenolic OH group is not coordinated to tellurium[79].
4. In addition to this a spectra of Schiff base ligand, band at 1629 cm^{-1} is due to vibration of azomethine group[90]. This band is shifted to higher and lower wavenumbers[81,91-93] (±4 cm^{-1}) in the complexes indicating the participation of the azomethine nitrogen in coordination[94].
5. New bands are found in spectra of complexes in the region 280-295 cm^{-1}, which are assigned to ν_{Te-O} stretching vibration[94-97] for Schiff base tellurium complexes. The bands at 427-450 cm^{-1} in complexes have been assigned to ν_{Te-N} of the azomethine mode[98].

From IR studies, it is conclude that Schiff base behave as a uninegative bidentate ligand with *-N, -O* donor sites coordinating to organyltellurium(IV) chloride *via* azomethine *N* and deprotonated phenolic -O atom.

^{1}H NMR spectra

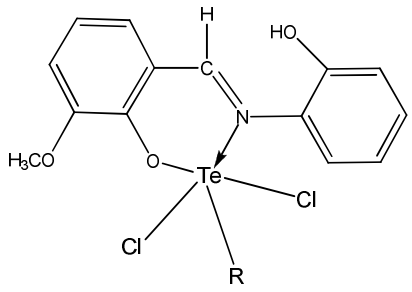
The proton chemical shift data of Schiff base and its complexes are given in Table 3.

1. The OH signals appeared in the spectrum of Schiff base at 14.15 δ ppm and 9.66 δ ppm are attributed to phenolic OH group present in *o*-vanillinidene part[83,99,100] and OH group present in the aminophenol moiety[99] indicates strong intramolecular hydrogen bonding[83]. The absence of the peak at 14.15 δ ppm in all complexes is as a result of enolization followed by deprotonation[83,101-103], indicates phenolic OH group of *o*-vanillinidene takes part in coordination[102]. Presence of signal of OH group which resonate at 10.21-9.82 δ ppm in complexes shows the OH group of aminophenol does not take part in coordination[104] which is also predicted by IR data.
2. The single signal of methyl group at δ 3.84 ppm of methoxy in *o*-vanillinidene part of Schiff base further split in the complexes indicating that the methyl group in ligand and complexes is not in identical environment, further[105] conclude that hydroxy group of *o*-vanillinidene moiety take part in coordination.
3. In Schiff base ligand the azomethine proton[106] resonate at 8.89 δ ppm which shift to downfield in complexes as compared to the free ligand, suggesting deshielding[99] of azomethine proton due to coordination[99,106] to metal ion through the azomethine nitrogen atom. On the basis of these studies, the proposed structures for the complexes are as below (Figure 2).

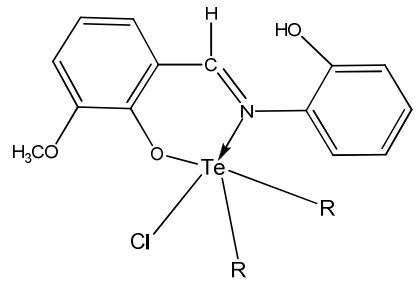
Table 3. ^{1}H NMR spectral data of Schiff Base and complexes in DMSO-d_6

Compound	Phenolic proton in *o*-vanillin moiety	Phenolic proton in 2-aminophenol moiety	Azomethine proton –N=CH	Benzene Ring proton	–OCH_3 proton on *o*-vanillin moiety
o-$VAPH_2$	14.15 s	9.64 s	8.89 s	6.80-8.39 m	3.84 s
1	-	9.74 s	8.93 s	6.32-8.28 m	3.83 t
2	-	9.91 s	8.91 s	6.31-8.28 m	3.85 t
3	-	9.72 s	8.91 s	6.80-8.15 m	3.84 t
4	-	9.71 s	8.92 s	6.82-8.11 m	3.44 m
5	-	10.18 s	9.96 s	6.75-8.88 m	3.84 m
6	-	9.68 s	8.90 s	6.75-7.90 m	3.99 m
7	-	10.18 m	8.88 s	6.30-8.10 m	3.84 m
8	-	9.82 s	8.94 s	6.12-8.10 m	3.76 s

s=singlet, t=triplet, m=multiplet



(*o*-VAPH).$RTeCl_2$



(*o*-VAPH).R_2TeCl

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

Figure 2. Proposed structures of complexes

Biological activity

The Schiff base (*o*-$VAPH_2$) and newly synthesized organyltellurium(IV) Schiff base complexes were screened for their *in vitro* antimicrobial potential against Gram +ve bacteria: *S.aureus* ATCC 11632 and *B.cereus* MTCC 7350, Gram -ve bacteria *E.coli* ATCC 35218, *P.aeruginosa* ATCC 23564, *S.typhi* ATCC 15499 and *P.rettgeri* DRDE strain; fungal strains *A.niger, A.fumigates* and *A.flavus* by tube dilution method[107]. Dilution of test and standard compounds were prepared Double strength nutrient broth- I.P (Antibacterial) and Sabouraud Dextrose Broth –I.P (Antifungal)[108]. The samples were incubated at 37±1 ºC for 24 h (bacteria), 25±1 ºC for 7 days (*A.niger*), 30±1 ºC for 15 days (*A.flavus*), 35±1 ºC for 72 h (*A.fumigates*) respectively and results were recorded in terms of MIC values are presented in the Table 4.

Table 4. Minimum inhibitory concentration, MIC, µg/mL; (-) Resistant

Compound	Bacterial strains						Fungal strains		
	S. aureus (ATCC 11632)	*S. typhi* (ATCC 15499)	*P. aeruginosa* (ATCC 23564)	*E. coli* (ATCC 35218)	*B. cereus* (MTCC 7350)	*P. rettgeri* (DRDE strain)	*A. niger*	*A. fumigates*	*A. flavus*
o-$VAPH_2$	1.25	-	5.0	1.25	0.625	-	-	10	-
1	5	10	5	20	-	0.625	-	-	-
2	20	-	-	-	20	-	-	-	10
3	-	-	5	1.25	0.625	-	10	10	-
4	10	10	20	-	5	5	5.0	-	-
5	-	-	5	1.25	0.625	-	20	5.0	1.25
6	20	-	20	-	10	20	-	-	2.5
7	20	-	20	-	10	20	-	1.25	5.0
8	10	10	20	-	5	5	5.0	10	5.0

Comparative study of the MIC value for Schiff base (*o*-$VAPH_2$) and their tellurium(IV) complexes indicates that the complexes exhibit higher antifungal activity than Schiff base itself. It has been also observed that the complexes show less antibacterial activity than Schiff base itself except 4-methoxytellurium(IV) complexes which show stronger activity against *P.rettgeri* but Schiff base does not show any activity against *P.rettgeri*.

Conclusion

o-$VAPH_2$ has been prepared by condensation of the Schiff base *o*-vanillin with 2-aminophenol. Sodium salt of this Schiff base when reacted with organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides in 1:1 molar ratios yield *o*-$VAPH.RTeCl_2$ and *o*-$VAPH.R_2TeCl$ (R=4-methoxyphenyl, 4-ethoxyphenyl, 4-hydroxyphenyl and 3-ethyl-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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