DOI:10.7598/cst2017.1394
ISSN:2278-3458

Chemical Science Transactions
2017, **6(3),** 448-456

RESEARCH ARTICLE

New Aryltellurium(IV) Complexes Containing NO Donor Schiff Base Derived from Vanillin and 4-Aminoantipyrine

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Received 22 February 2017 / Accepted 2 March 2017

Abstract: A novel bidentate Schiff base derived from vanillin and 4-aminoantipyrine, form 1:1 complexes with aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides of the type VL-AAP.ArTeCl$_3$ and VL-AAP.Ar$_2$TeCl$_2$ (where Ar = *p*-methoxyphenyl, *p*-ethoxyphenyl, *p*-hydroxy-phenyl and 3-methyl-4-hydroxyphenyl and VL-AAP =Schiff base). These complexes have been characterized by elemental analyses, molar conductance, IR and ^{1}H NMR spectroscopy. On the basis of these studies the donor sites have been identified as carbonyl oxygen of the pyrazolone ring and nitrogen atom of azomethine group (CH=N) to give five membered chelate ring with tellurium centre having Ψ-octahedral geometry. The synthesized ligand and its aryltellurium complexes were screened for their antimicrobial activity against various bacterial and fungal strains.

Keywords: Vanillin, 4-Aminoantipyrine, Aryltellurium(IV), Diaryltellurium(IV), Antimicrobial activity

Introduction

In the last few decades, there has been a considerable interest in the chemistry of antipyrine and its derivative due to their potential biological activity[1] as analgesic[2,3] and anti-inflammatory[3], antiviral[4,5], antibacterial[4,5] and anticancer activity[6].

Among the various derivatives, 4-aminoantipyrine form a variety of Schiff bases with aldehydes/ketones and are reported to be effective ligands having biological and clinical applications[7-9]. The ligands contain antipyrine skeleton in their structure having the hetroatoms in the ring[10] which induces the biological activity[11-13]. The carbonyl group in the base is a potential donor due to large dipole moment[14] (5.48 D) and strong basic character.

Also, aryltellurium(IV) chlorides are known to act as Lewis acids[15-31] and form complexes with several N-, O- and S- donor bases. As a part of our continuing interest on the synthesis of potential bioactive compounds, we hereby report the synthesis, characterization and biological studies on aryltellurium(IV) trichlorides, ArTeCl$_3$ and diaryltellurium(IV) dichlorides, Ar$_2$TeCl$_2$ complexes with Schiff base derived from 4-aminoantipyrine and vanillin.

Experimental

All preparations were carried out under dry nitrogen atmosphere and the solvents used were purified by standard method[32,33] 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 dip type conductivity cell on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra (4000-400 cm^{-1}) 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 tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer. The antimicrobial activity was evaluated by tube dilution method at Department of Pharmaceutical Sciences, M. D. University, Rohtak,India.

Preparation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides

p-Methoxyphenyltellurium(IV) trichloride[34,35], bis(*p*-methoxyphenyl)tellurium(IV) dichloride[35,36], *p*-ethoxyphenyltellurium(IV) trichloride[37], bis(*p*-ethoxyphenyl)tellurium dichloride[37] *p*-hydroxyphenyltellurium(IV) trichloride[38], bis(*p*-hydroxyphenyl)tellurium(IV) dichloride[38], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride[39] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride[39] were prepared by the reactions of $TeCl_4$ with anisole, phenetole, phenol, *o*-cresol respectively, by the methods reported in the literature[34-39].

Preparation of vanillin-4-aminoantipyrine Schiff base (VL-AAP)[40]

Equimolar amounts of saturated methanolic solution of vanillin and 4-aminoantipyrine were mixed thoroughly and few drops of glacial acetic acid were added. The mixture was refluxed for 4 hours at 70-80 ˚C on a water bath. The resulting solution was cooled at room temperature and then poured into crushed ice with constant stirring. The sodium bisulphite solution was added to the precipitate to remove excess aldehyde if any. The crystalline product was dried under *vacuum* and kept in desiccator over P_4O_{10} until further use. Yield = 90%, M.pt.=165-167 ^oC. Analysis (Calculated) $C_{19}H_{19}N_3O_3$:C(67.66), H(5.68) and N(12.46); Found: C(67.50), H(5.82) and N(12.27)

Preparation of Schiff base complexes of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides

Aryltellurium(IV) trichlorides, $ArTeCl_3$ and diaryltellurium(IV) dichlorides Ar_2TeCl_2 (Ar=*p*-methoxyphenyl, *p*-ethoxyphenyl, *p*-hydroxyphenyl and 3-methyl-4-hydroxyphenyl), when reacted with VL-AAP in equimolar ratio, yield VL-AAP.$ArTeCl_3$ and VL-AAP.Ar_2TeCl_2 type complexes.

A saturated methanolic solution of 2 mmol of aryltellurium(IV) trichloride or diaryltellurium(IV) dichloride was added dropwise to the suspension of 2 mmol Schiff base in 60 mL benzene + 40 mL methanol. The reaction mixture was further refluxed for 3-4 h, cooled and filtered off to remove any turbidity. 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}.

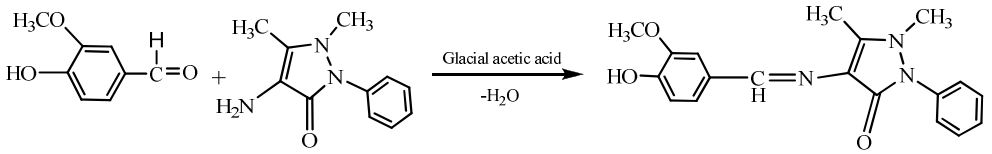
Results and Discussion

$TeCl_4$ when heated with anisole[34-36], phenetole[37], phenol[38], *o*-cresol[39] (Ar-H) appears to undergo Friedel-Crafts type condensation reaction whereby $TeCl_3^+$ unit attacks a position *para* to the alkoxy/hydroxyl groups in the aromatic rings, thus resulting in the formation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides.

$$Ar\text{-}H + TeCl_4 \longrightarrow ArTeCl_3 + HCl \quad (1)$$

$$2\ Ar\text{-}H + TeCl_4 \longrightarrow Ar_2TeCl_2 + 2\ HCl \quad (2)$$

Formation of Schiff base (VL-AAP) by the reaction of vanillin drug and 4-aminoantipyrine can be represented by following equations.



Vanillin 4-aminoantipyrine Schiff base(VL-AAP)

Schiff base (VL-AAP) reacts with aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides in 1:1 molar ratio to yield the corresponding aryltellurium(IV) complexes.

$$VL\text{-}AAP + ArTeCl_3 \longrightarrow (VL\text{-}AAP)\ ArTeCl_3 \quad (3)$$

$$VL\text{-}AAP + Ar_2TeCl_2 \longrightarrow (VL\text{-}AAP)\ Ar_2TeCl_2 \quad (4)$$

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

Conductance studies

Molar conductance (Λ_M) data for the complexes at *ca.* 10^{-3} M in DMSO (Table 1) lie in the range 12.41-25.22 S cm^2 mol^{-1}, which predict the non electrolyte[41,42] type behaviour of these complexes in DMSO.

Infrared spectra

The IR data of Schiff base and its tellurium(IV) complexes are listed in Table 2. The spectra of VL-AAP Schiff base complexes are quite complex and an attempt has therefore been made to identify the donor sites by comparing the spectra of complexes with parent ligand and $ArTeCl_3/Ar_2TeCl_2$.

In IR spectrum of ligand shows intense band at 1621 cm^{-1} ascribed to carbonyl $\nu_{(C=O)}$ of the pyrazolone ring[40,43-47] has been shifted towards higher wavenumber in spectra of complexes indicating the linkage between the tellurium atom and carbonyl oxygen atom[43,45,46]. The band at 1581 cm^{-1} for the azomethine[40,43,45] group of the Schiff base shifted to higher frequency in the IR spectra of aryltellurium complexes due to the bonding of the azomethine nitrogen to tellurium atom. This may be mixed with $\nu_{(C=C)}$ of the $ArTe/Ar_2Te$ moieties. Also, a broad band in Schiff base around 3176 cm^{-1} indicates the presence of phenolic[43,48] OH. This band is slightly shifted to around 3450 cm^{-1} in the Schiff base tellurium complexes, this shows that the hydroxyl group oxygen does not contribute in bonding pattern[48].

Table1. Analytical data, molar conductance and physical properties of Schiff base (VL-AAP) complexes of tellurium(IV)

Compound No.	Complex (Ar)	Empirical formula (Formula Wt.)	Colour (Yield, %)	M. Pt. (°C) dec.	Analyses % Found (Calculated)					Λ_M at *ca.* 10^{-3} M Scm^2mol^{-1} in DMSO
					C	H	N	Te	Cl	
Schiff Base	VL-AAP	$C_{19}H_{19}N_3O_3$	Yellow (90)	165-167	67.50 (67.66)	5.82 (5.68)	12.27 (12.46)	-	-	-
1	(VL-AAP).$ArTeCl_3$ (*p*-methoxyphenyl)	$C_{26}H_{26}Cl_3N_3O_4Te$ (678.46)	Greenish -yellow (85)	128-130	45.89 (46.03)	3.99 (3.86)	6.07 (6.19)	18.72 (18.81)	15.52 (15.68)	16.78
2	(VL-AAP).$ArTeCl_3$ (*p*-ethoxyphenyl)	$C_{27}H_{28}Cl_3N_3O_4Te$ (672.07)	Light yellow (88)	150-152	45.63 (46.83)	4.21 (4.08)	5.87 (6.07)	18.55 (18.43)	15.17 (15.36)	15.76
3	(VL-AAP).$ArTeCl_3$ (*p*-hydroxyphenyl)	$C_{25}H_{24}Cl_3N_3O_4Te$ (644.43)	Dark yellow (85)	132-134	45.07 (45.19)	3.52 (3.64)	6.15 (6.32)	19.05 (19.20)	15.89 (16.01)	19.74
4	(VL-AAP).$ArTeCl_3$ (3-methyl-4-hydroxyphenyl)	$C_{26}H_{26}Cl_3N_3O_4Te$ (678.46)	Light brown (82)	118-120	45.87 (46.03)	3.98 (3.86)	6.01 (6.19)	18.51 (18.81)	15.47 (15.68)	14.01
5	(VL-AAP). Ar_2TeCl_2 (*p*-methoxyphenyl)	$C_{33}H_{33}Cl_2N_3O_5Te$ (750.14)	Dark brown (85)	92-94	52.73 (52.84)	4.34 (4.43)	5.49 (5.60)	16.92 (17.01)	9.33 (9.45)	18.73
6	(VL-AAP). Ar_2TeCl_2 (*p*-ethoxyphenyl)	$C_{35}H_{37}Cl_2N_3O_5Te$ (778.19)	Red (82)	82-84	53.89 (54.02)	4.91 (4.79)	5.30 (5.40)	16.31 (16.40)	9.01 (9.11)	25.22
7	(VL-AAP).Ar_2TeCl_2 (*p*-hydroxyphenyl)	$C_{31}H_{29}Cl_2N_3O_5Te$ (722.09)	Brown (85)	102-104	51.47 (51.56)	4.15 (4.05)	5.53 (5.82)	17.55 (17.67)	9.65 (9.82)	12.41
8	(VL-AAP). Ar_2TeCl_2 (3-methyl-4-hydroxyphenyl)	$C_{33}H_{33}Cl_2N_3O_5Te$ (750.14)	Brown (70)	158-160	52.71 (52.84)	4.24 (4.43)	5.49 (5.60)	16.89 (17.01)	9.33 (9.45)	15.75

Values of Λ_M reported[41 42] for 1:1 electrolytes in DMSO=50-70 S cm^2 mol^{-1}

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

Compound	Phenolic $\nu_{(O-H)}$ of ligand	Carbonyl $\nu_{(C=O)}$ of pyrazolone	Azomethine $\nu_{(C=N)}$	$\nu_{(Te-N)}$	$\nu_{(Te-O)}$
VL-AAP	3176 mb	1621 s	1582 s	-	-
1	3451 mb	1637 s	1587 s	420 s	273 w
2	3452 mb	1634 s	1586 s	418 m	271 w
3	3448 mb	1637 s	1586 s	418 s	288 w
4	3445 mb	1635 s	1588 s	420 s	285 w
5	3448 mb	1625 s	1581 s	420 s	291 w
6	3420 mb	1624 sh	1582 s	425 m	277 w
7	3451 mb	1625 sh	1583 s	420 m	282 w
8	3448 mb	1632 sh	1586 s	415 m	287 w

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

The two new bands appear in range 281-293 cm^{-1} and 415-425 cm^{-1} assigned due to $\nu_{(Te-O)}$[49-52] and $\nu_{(Te-N)}$ mode[53] of vibration. Thus, IR data predict the bidentate nature of the Schiff base (VL-AAP) involving azomethine nitrogen atom and carbonyl oxygen of pyrazolone ring give rise to six membered chelate rings with the tellurium centre having distorted octahedral geometry.

^{1}H NMR spectra

In order to identify the solution structure of Schiff base (VL-AAP) and its complexes, ^{1}H NMR spectra were recorded in DMSO-d_6 and the data are given in Table 3. The proton signal for OH group[54] in Schiff base appears[44,45,47] at 9.49 δ ppm as broad singlet and in complexes it resonate at 9.76 δ ppm. This indicates that OH group does not participate in the complexation[45].

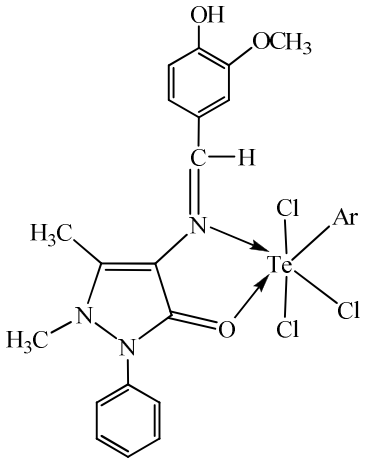
Table 3. ^{1}H NMR spectral data of Schiff base (VL-AAP) and complexes in DMSO-d_6

Compound	Phenolic-OH of ligand	Azomethine-H	*N*-Methyl proton of pyrazolone ring	*C*-Methyl proton of pyrazolone ring
VL-AAP	9.495 bs	9.461 s	3.451 m	2.445 m
1	9.761 s	9.487 s	3.829 m	2.537 m
2	9.762 s	9.493 s	3.871 m	2.533 m
3	9.763 s	9.356 s	3.910 m	2.560 m
4	9.763 s	9.382 s	3.863 m	2.536 m
5	9.762 s	9.484 s	3.884 m	2.560 m
6	9.764 s	9.477 s	3.861 m	2.526 m
7	9.761 s	9.474 s	3.864 m	2.528 m
8	9.780 s	9.385 s	3.868 m	2.531 m

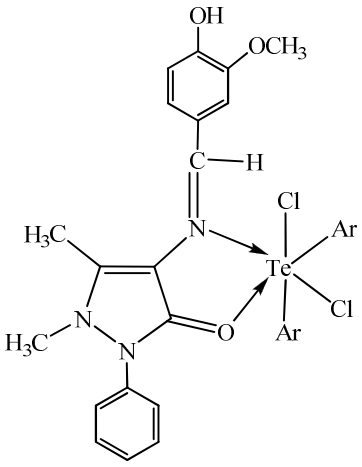
bs=broad singlet s=singlet, m=multiplet

The azomethine protons which resonate as a singlet[43,44,46] at 9.47 δ ppm, the coordination of azomethine nitrogen to tellurium in the complexes are clearly demonstrated by a downfield shift of this peak[43]. The signal for *C*-methyl proton (>C-CH_3) attached to pyrazolone ring[43,46] appear at 2.44 δ ppm, while the *N*-methyl proton (>N-CH_3) attachéd to pyrazolone[43,46] appear at 3.45 δ ppm in the spectra of Schiff base and these signal shift to down field in complexes due to the effect of coordination of carbonyl oxygen. The ratios of ligand protons to the ArTe/ Ar_2Te protons also confirm the stoichiometry of complexes as 1:1.

Thus, Schiff base (VL-AAP) act as a bidentate-*N*(azomethine), -*O*(carbonyl) chelating ligand in VL-AAP.$ArTeCl_3$ and VL-AAP.Ar_2TeCl_2 complexes giving six coordinate tellurium having Ψ-octahedral geometry in these complexes as predicated from IR studies as well. The proposed structures are as given below (Figure 1).



VL-AAP.$ArTeCl_3$



VL--AAP.Ar_2TeCl_2

Ar = *p*-methoxyphenyl, *p*-ethoxyphenyl, *p*-hydroxyphenyl and 3-methyl-4-hydroxyphenyl

Figure 1. Proposed structures of complexes

Biological activity

The Schiff base (VL-AAP) and newly synthesized aryltellurium(IV) schiff base complexes were evaluated for their antimicrobial activity *in vitro* 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) and fungal strains (*A.niger, A.fumigates* and *A.flavus*) by tube dilution method[55]. Dilution of test and standard compounds were prepared double strength nutrient broth- I.P (Antibacterial) and sabouraud

dextrose broth -I.P (Antifungal)[56]. 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. The lowest concentration of test substances which inhibited 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*
1	5.0	10	5	20	-	0.625	10	10	-
2	1.25	2.5	1.25	5.0	-	-	-	-	-
3	5.0	-	-	-	1.25	2.5	5.0	-	-
4	-	-	5.0	1.25	0.625	-	-	1.25	5.0
5	5.0	10	5.0	20	-	0.625	20	5.0	1.25
6	10	10	20	-	5.0	5.0	5.0	2.5	-
7	20	-	20	-	10	20	-	-	2.5
8	-	5.0	1.25	-	-	-	-	0.625	-

Comparative study of the MIC value for Schiff base (*o*-VAPH) and their tellurium(IV) complexes indicates that the complexes shows higher antimicrobial (bacterial and fungal) activity than Schiff base itself. From the data, the antibacterial activity shows following trend:

$$VL\text{-}AAP.ArTeCl_3 > VL\text{-}AAP.Ar_2TeCl_2 > VL\text{-}AAP \text{ Schiff base}$$

And for antifungal activity trend:

$$VL\text{-}AAP.Ar_2TeCl_2 > VL\text{-}AAP.ArTeCl_3 > VL\text{-}AAP \text{ Schiff base}$$

Conclusion

Aryltellurium(IV) and diaryltellurium(IV) dichlorides upon reaction with Schiff base (VL-AAP) derived from vanillin and 4-aminoantipyrine yield new 1:1 type complexes of tellurium(IV). The synthesized complexes were characterized by elemental analyses, conductance measurement, IR and ^{1}H NMR spectral studies. The analytical data suggest that the VL-AAP Schiff base complexes have 1:1 stoichiometry. The Schiff base (VL-AAP) in these complexes acts as a bidentate ligand through oxygen of the pyrazolone ring carbonyl (C=O) and nitrogen atom of azomethine group (CH=N) to give five membered chelate ring with tellurium centre having Ψ-octahedral geometry. The complexes have been observed to possess more antimicrobial activity against bacterial and fungal strains than parent Schiff base.

Acknowledgement

The authors are grateful to M. D. University, Rohtak for providing the necessary facilities. One of the authors (Deepak) is also thankful to UGC New Delhi for providing fellowship. We also thank SAIF, Panjab University Chandigarh for providing the CHN analyses and spectral data.

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