

Spectroscopic and Biological Studies of 3,5-Dichlorosalicylaldehyde-3-Aminopyridine Schiff Base and their Tellurium(IV) Complexes

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ABSTRACT

3,5-Dichlorosalicylaldehyde react with 3-aminopyridine to gives monobasic ON bidentate Schiff base ligand (3-APY-{3,5-ClSalH}). TeCl_4 , ArTeCl_3 and Ar_2TeCl_2 : where Ar = 4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl, react with above Schiff base leads to seven new complexes: having formula $[\text{TeCl}_3(3\text{-APY}\{-3,5\text{-ClSal}\})]$, $[\text{ArTeCl}_2(3\text{-APY}\{-3,5\text{-ClSal}\})]$ and $[\text{Ar}_2\text{TeCl}(3\text{-APY}\{-3,5\text{-ClSal}\})]$. The purity of these compounds was estimated by TLC technique, while their structures were supported by elemental analyses, conductance measurement and spectroscopic methods such as $^1\text{H NMR}$, infrared spectra giving pentacoordinated tellurium centre having Ψ – trigonal bipyramidal geometry. Also, Schiff base ligand and some of their tellurium(IV) complexes display biological activities in vitro against bacteria and fungi.

Keywords: 3,5-Dichlorosalicylaldehyde, Bidentate Schiff base, Tellurium(IV) Complexes.

I. INTRODUCTION

Schiff bases are considered as “privileged ligands” because they are easily prepared by the condensation product of carbonyl groups with amine[1-6]. Some Schiff bases containing nitrogen and other donor atoms are reported to possess antibacterial[1,7-12], antifungal[9-13] and antitumor activities[14,15]. It is reported[1,16] Schiff base containing heteroatom[17,18] and 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 activity[19-30].

TeCl_4 is also known to form adducts with amides[31-38] and thiourea[39], thus reflecting its acceptor behaviour. Also, aryltellurium(IV) chlorides are known[31-36,40-57] to behave as Lewis acids and

form complexes with several Nitrogen, Oxygen and Sulphur donor bases. In view of this, we have investigated the reactions of tellurium(IV) chloride and aryltellurium(IV) chlorides with 3,5-dichlorosalicylaldehyde-3-aminopyridine Schiff base (3-APY-{3,5-ClSalH}), to synthesize some new complexes of tellurium(IV).

II. METHODS AND MATERIAL

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[58,59] 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 $25 \pm 2^\circ\text{C}$ with a dip type conductivity cell on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra ($4000\text{--}40\text{ cm}^{-1}$) 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- d_6 using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer from CIL, Guru Jambheshwar University of Science and Technology, Hissar and the Microcare Laboratory, Surat, for providing antimicrobial activities.

Preparation of Aryltellurium(IV) Trichlorides and Diaryltellurium(IV) Dichlorides

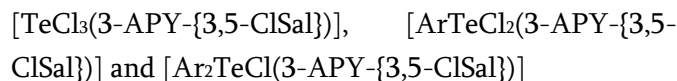
4-Methoxyphenyltellurium(IV) trichloride[60,61], bis(p-methoxyphenyl)tellurium(IV) dichloride[61,62], 4-hydroxyphenyltellurium(IV) trichloride[63], bis(p-hydroxyphenyl)tellurium(IV) dichloride[63], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride[64] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride[64] 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[60-64].

Preparation of 3,5-Dichlorosalicylaldehyde-3-Aminopyridine Schiff base

Equimolecular quantity of 3,5-Dichlorosalicylaldehyde (0.08 mole, 15.29g) and 3-aminopyridine (0.08 mole, 7.52g) were mixed in 25 ml methanol in a round bottomed flask equipped with a condenser[65]. The reaction mixture was refluxed on waterbath for 4 hours. After cooling, the precipitated Schiff base was collected by filtration and recrystallized from methanol. The light yellow colour product was dried in a desiccator over anhydrous CaCl_2 and kept over P_4O_{10} . Yield = 95%

Preparation of Complexes

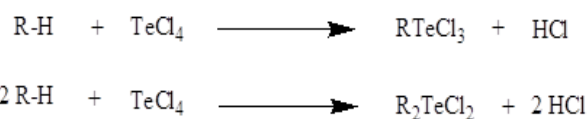
Tellurium tetrachloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides, when reacted with Schiff base to form solid complexes as described below:



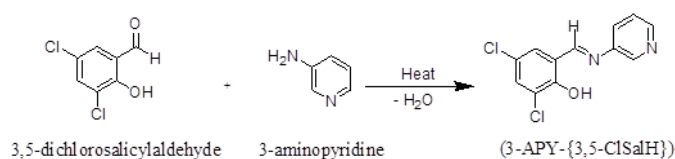
The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL anhydrous methanol to a hot solution of 5 mmol Schiff base (3-APY-{3,5-ClSalH}) in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent 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 P_4O_{10} .

III. RESULTS AND DISCUSSION

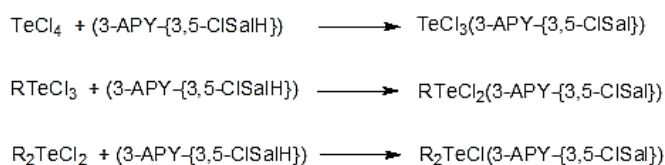
When Anisole[60-62], phenol[63] and o-cresol[64] (R-H) heated with TeCl_4 appears to undergo the Friedel Craft type condensation reaction where by TeCl_3^+ unit attacks a position para to the methoxy / hydroxyl group in the aromatic ring, thus resulting in the formation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides.



Preparation of Schiff Base by the reaction of 3,5-Dichlorosalicylaldehyde with 3-aminopyridine can be represented by following equation.



Schiff Base reacts with tellurium (IV) chloride, aryltellurium (IV) trichlorides and diaryltellurium (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 aryltellurium(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 21.45-51.08 S $\text{cm}^2 \text{mol}^{-1}$ which predict the non-electrolyte to 1:1 weak electrolyte type behavior[66,67] of these Schiff base complexes in DMSO, probably due to ionization into $\text{TeCl}_2(3\text{-APY-}\{3,5\text{-ClSal}\})^+ / \text{ArTeCl}(3\text{-APY-}\{3,5\text{-ClSal}\})^+ / \text{Ar}_2\text{Te}(3\text{-APY-}\{3,5\text{-ClSal}\})^+$ 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- $\{3,5\text{-ClSal}\}^-$ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes[68] which are reported to be non-electrolytes.

Infrared Spectra

The assignment of the important bands was made and recorded in Table 2. The vibrational spectra of the ligand exhibit the characteristic bands[2,37,65,69],

one at 1621 cm^{-1} of azomethine $\nu_{(\text{C}=\text{N})}$ group and another at 3054 cm^{-1} , of phenolic $\nu_{(\text{OH})}$ group. While the IR spectra of tellurium complexes show sharp bands in the range $1605\text{-}1613 \text{ cm}^{-1}$ which is shifted to lower frequency region[2,13,65,69-71] as compared to ligand i.e. 1621 cm^{-1} suggesting a decrease in the C=N bond strength due to the formation of coordinate bond of the metal with the imine nitrogen lone pair[72,73]. The disappearance of phenolic -OH broad peak in the complexes shows the deprotonation of -OH group and its subsequent coordinatyon to the central metal atom.

Two new bands observed at $282\text{-}290 \text{ cm}^{-1}$ due to $\nu_{(\text{Te}-\text{O})}$ [70,71,74,75] and $419\text{-}420 \text{ cm}^{-1}$ due to $\nu_{(\text{Te}-\text{N})}$ [76] absorptions respectively, further support the involvement of phenolic oxygen and azomethine nitrogen atom of Schiff base in the coordination.

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.

¹H NMR Spectra

¹H NMR spectrum of Schiff base and its complexes were recorded in DMSO- d_6 and compiled in Table 3. Schiff base characterized by three signals appeared singlet at 13.632δ ppm assigned to phenolic -OH and 9.712δ ppm which is assigned to azomethine proton -N=CH- whereas signal due to aromatic moiety appeared in the range $7.263\text{-}8.609$ (complex multiplet) δ ppm.

The signal due to azomethine proton in the complexes i.e. $[\text{TeCl}_3(3\text{-APY-}\{3,5\text{-ClSal}\})]$, $[\text{ArTeCl}_2(3\text{-APY-}\{3,5\text{-ClSal}\})]$ and $[\text{Ar}_2\text{TeCl}(3\text{-APY-}\{3,5\text{-ClSal}\})]$, shows downfield shifting as compared to free ligand, suggesting deshielding of azomethine proton due to coordination to tellurium through the azomethine nitrogen[48,49,69,77].

Table 1. Analytical Data, Molar Conductance and Physical Properties for Schiff Base (3-APY-{3,5-ClSalH}) Complexes of Tellurium(IV)

Compound 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-{3,5-ClSalH})	$C_{12}H_8Cl_2N_2O$ (267.14)	Light Yellow (95)	149-151	53.78 (53.95)	3.07 (2.99)	10.36 (10.49)	-	26.22 (26.58)	-
I	$TeCl_3(3-APY-\{3,5-ClSal\})$	$C_{12}^7H_8^5Cl_2^2N_2^2O$ (500.24)	Reddish Brown (82)	240-242	28.59 (28.81)	1.28 (1.40)	5.52 (5.60)	29.24 (29.58)	35.27 (35.48)	26.90
II	$ArTeCl_2(3-APY-\{3,5-ClSal\})$ (4-methoxyphenyl)	$C_{19}^4H_{14}^4Cl_2^2N_2^2O$ (571.81)	Light Yellow (75)	195-197	38.68 (39.91)	2.26 (2.45)	4.81 (4.90)	24.99 (25.37)	24.34 (24.83)	29.76
III	$ArTeCl_2(3-APY-\{3,5-ClSal\})$ (4-hydroxyphenyl)	$C_{18}^4H_{12}^4Cl_2^2N_2^2O$ (557.80)	Brown (81)	234-236	38.59 (38.76)	1.99 (2.15)	4.93 (5.02)	25.84 (26.10)	25.09 (25.46)	36.07
IV	$ArTeCl_2(3-APY-\{3,5-ClSal\})$ (3-methyl-4-hydroxyphenyl)	$C_{19}^4H_{14}^4Cl_2^2N_2^2O$ (571.81)	Light Brown (83)	210-212	38.68 (39.91)	2.26 (2.45)	4.81 (4.90)	24.99 (25.37)	24.34 (24.83)	21.45
V	$Ar_2TeCl(3-APY-\{3,5-ClSal\})$ (4-methoxyphenyl)	$C_{26}^3H_{21}^3Cl_3^3N_2^3O$ (643.38)	Light Orange (70)	221-223	48.43 (48.53)	3.19 (3.26)	3.99 (4.36)	21.99 (22.21)	16.11 (16.55)	46.89
VI	$Ar_2TeCl(3-APY-\{3,5-ClSal\})$ (4-hydroxyphenyl)	$C_{24}^3H_{17}^3Cl_3^3N_2^3O$ (615.36)	Cream (72)	214-216	46.70 (46.84)	2.60 (2.76)	4.23 (4.55)	23.02 (23.35)	16.95 (17.31)	51.08
VII	$Ar_2TeCl(3-APY-\{3,5-ClSal\})$ (3-methyl-4-hydroxyphenyl)	$C_{26}^3H_{21}^3Cl_3^3N_2^3O$ (643.38)	Pinkish brown (77)	228-230	48.43 (48.53)	3.19 (3.26)	3.99 (4.36)	21.99 (22.21)	16.11 (16.55)	49.05

Values of Λ_M reported[66,67] for 1:1 electrolytes in DMSO = 50 – 70 $S\ cm^2\ mol^{-1}$

Table 2. Important IR Data (cm^{-1}) of the Schiff Base (3-APY-{3,5-ClSalH}) and complexes.

Compound No.	(Phenolic) ν (OH)	(Azomethine) ν (C=N)	ν (Te-O)	ν (Te-N)
(3-APY-{3,5-ClSalH})	3054 b	1621 s	-	-
I	-	1608 s	290 s	419 s
II	-	1605 s	282 s	420 s
III	-	1607 s	282 s	419 s
IV	-	1609 s	289 s	419 s
V	-	1606 sh	290 s	419 s
VI	-	1613 s	290 s	419 s
VII	-	1607 s	289 s	419 b

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

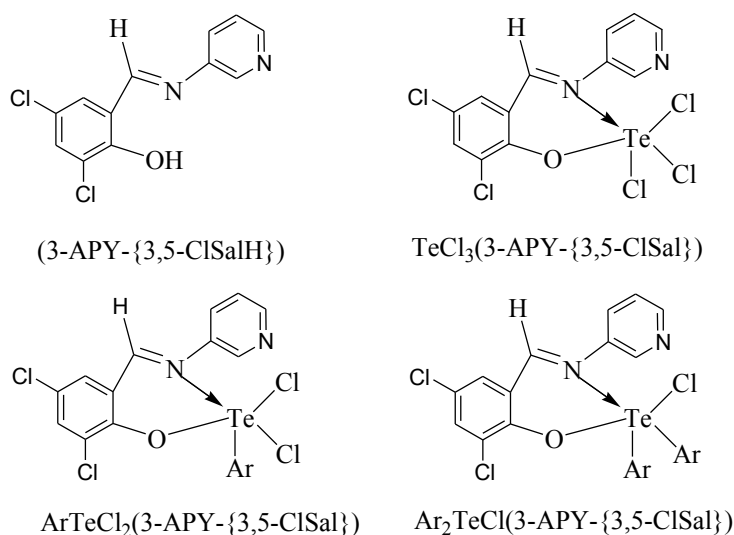
Another singlet at 13.632 δ ppm due to phenolic -OH group disappeared in the complexes confirms the participation of -OH group of 3,5-dichlorosalicylaldehyde in the coordination via deprotonation.

On the basis of spectral studies, it may be concluded that (3-APY-{3,5-ClSalH}) acts as uninegative (ON) bidentate ligand, resulting in the formation of pentacoordinated tellurium(IV) complexes and proposed structures are shown in Figure 1.

Table 3. ^1H NMR Spectral Data of Schiff Base (3-APY-{3,5-ClSalH}) and Complexes in DMSO- d_6 .

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-{3,5-ClSalH})	13.632 (s, 1H)	9.712 (s, 1H)	7.263-8.609 (cm, 6H)	-	-
I	-	10.146 (s, 1H)	7.646-8.545 (cm, 6H)	-	-
II	-	10.141 (s, 1H)	7.038-8.352 (cm, 10H)	3.809 (s,3H*)	-
IV	-	10.140 (s, 1H)	6.837-8.235 (cm, 9H)	2.512 (s,3H)	9.088 (s,1H)
V	-	10.152 (s, 1H)	7.622-8.020 (cm, 14H)	3.379 (s,6H*)	-
VI	-	10.137 (s, 1H)	6.837-8.235 (cm, 14H)	-	9.086 (s,2H)
VII	-	10.139 (s, 1H)	6.866-8.001 (cm, 12H)	3.402 (s,6H)	9.088 (s,2H)

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

**Figure 1:** Proposed Structures of Schiff base (3-APY-{3,5-ClSalH}) and Tellurium (IV) Complexes.

Biological Studies

Schiff base and all newly synthesized complexes were tested 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 activities[78] are given in Table 4.

The data shows that the complex no. II i.e. [RTeCl₂(3-APY-{3,5-ClSal})]: where R = 4-hydroxyphenyl, shows good activity against *Escherichia coli* bacterial strain than Schiff base itself. Most of the complexes are moderately more effective against bacterial strain *Staphylococcus aureus* MTCC 96 as compare to free ligand.

Table 4. Minimum Inhibitory Concentration MIC ($\mu\text{g/mL}$) of Schiff Base (3-APY-{3,5-ClSalH}) 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-{3,5-ClSalH})	500	500	200	200	1000	500	500
I	250	250	200	200	500	> 1000	> 1000
II	200	200	200	125	1000	> 1000	> 1000
III	250	500	250	250	1000	500	1000
VI	500	500	500	500	> 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

IV. CONCLUSION

The Schiff base has been prepared by condensation of 3,5-dichlorosalicylaldehyde with 3-aminopyridine. Schiff base when reacted with tellurium tetrachloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides in 1:1 molar ratios yield $[\text{TeCl}_3(3\text{-APY}\{-3,5\text{-ClSal}\})]$, $[\text{ArTeCl}_2(3\text{-APY}\{-3,5\text{-ClSal}\})]$ and $[\text{Ar}_2\text{TeCl}(3\text{-APY}\{-3,5\text{-ClSal}\})]$: where Ar = 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.

V. REFERENCES

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