International Journal of Advanced Science and Research
ISSN: 2455-4227
Impact Factor: RJIF 5.12
www.allsciencejournal.com
Volume 3; Issue 1; January 2018; Page No. 123-128

Synthesis and antimicrobial activities of Te (IV) complexes of 3, 5-dichlorosalicylaldehyde-2-aminopyridine schiff base

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Abstract

In the present study, Schiff base was prepared using 2-Aminopyridine with 3, 5-dichlorosalicylaldehyde and after that tellurium (IV) complexes were synthesized using the Schiff base. The structures of the newly synthesized complexes and Schiff base were analyzed by elemental analyses, conductivity measurements, FT-IR and proton NMR spectral studies. The tellurium(IV) complexes are distorted octahedrally coordinated by phenolic oxygen as well as azomethine and pyridine nitrogen atoms to give rise the formula of complexes is **[**$TeCl_3$(2-APY-{3,5-ClSal})], [$RTeCl_2$(2-APY-{3,5-ClSal})] and [R_2TeCl(2-APY-{3,5-ClSal})]: where R = 4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl. Also, the Schiff base and some of their tellurium (IV) complexes were tested in vitro to evaluate *antifungal* and *antibacterial* activities.

Keywords: azomethine, 3, 5-dichlorosalicylaldehyde, schiff base, 2-aminopyridine, antifugal activity, antibacterial activity

1. Introduction

The compounds containing an azomethine group (-CH=N) are the condensation product of carbonyl groups (aldehydes or ketones) with amine [1, 2]. Schiff base ligands are considered to be good chelating agents and –OH functional group close to azomethine group made them as special class of ligands[3-6]. Due to the great flexibility and diverse structural aspects, wide ranges of Schiff base have been synthesized and their complexation behaviors have been studied. Antibacterial [1, 7-12], Antifungal [9-13] and Antitumor activities [14, 15] of Schiff base have been reported. Schiff bases having hetroatom16 and derived from substituted *o*-hydroxybenzaldehyde, *p*-hydroxybenzaldehyde and *p*-nitrobenzaldehyde etc. exhibit more antimicrobial activity [17-30].

Tellurium (IV) chloride is also known to form adducts with amides [31, 38] and thiourea [39], thus reflecting its acceptor behaviour. Also, organyltellurium (IV) chlorides are known [31-36, 1-57] to behave as lewis acids and form complexes with several N-, O- and S- donor bases. In connection with such studies, the present paper reporting for the first time the reactions of tellurium (IV) chloride and organyltellurium (IV) chlorides with 3,5-dichlorosalicylaldehyde-2-aminopyridine Schiff base (2-APY-{3,5-ClSalH}), to synthesize some new complexes of tellurium (IV).

2. Materials and Methods

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±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-d_6 using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer from CIL, Guru Jambeshwar University of Science and Technology, Hissar, Haryana, India.

Preparation of Organyltellurium (IV) Trichlorides and Diorganyltellurium (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-2-Aminopyridine Schiff base

The Schiff base was prepared by mixing equimolecular quantity of 3,5-Dichlorosalicylaldehyde (0.08 mole, 15.29g) and 2-aminopyridine (0.08 mole, 7.52g) in 10 ml methanol in a round bottomed flask equipped with a condenser[65]. The reaction mixture was refluxed on waterbath for 4 hours. After completion of reaction, the reaction mixture was cooled, filtered and dried in a desiccator over anhydrous $CaCl_2$ and recrystallized from methanol, a sharp orange crystalline product was obtained.

Preparation of Complexes

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

[$TeCl_3$(2-APY-{3,5-ClSal})], [$RTeCl_2$(2-APY-{3,5-ClSal})] and [R_2TeCl(2-APY-{3,5-ClSal})]

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 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}.

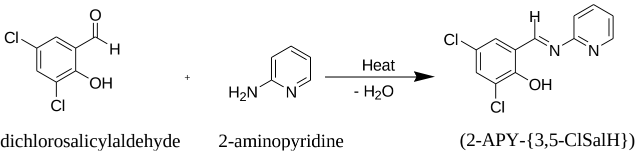
3. Results & Discussion

$TeCl_4$ when heated with anisole [60, 62], phenol [63] and *o*-cresol [64] (R-H) 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 organyltellurium (IV) trichlorides and diorganyltellurium (IV) dichlorides.

$$\text{R-H} + TeCl_4 \longrightarrow RTeCl_3 + HCl$$

$$\text{2 R-H} + TeCl_4 \longrightarrow R_2TeCl_2 + 2\,HCl$$

Preparation of Schiff Base by the reaction of 3, 5-dichlorosalicylaldehyde with 2-aminopyridine can be represented by following equation.



Schiff Base reacts with tellurium (IV) chloride, organyltellurium (IV) trichlorides and diorganyltellurium (IV) dichlorides to yield the coloured tellurium (IV) complexes.

$$TeCl_4 + \text{(2-APY-\{3,5-ClSalH\})} \longrightarrow TeCl_3\text{(2-APY-\{3,5-ClSal\})}$$

$$RTeCl_3 + \text{(2-APY-\{3,5-ClSalH\})} \longrightarrow RTeCl_2\text{(2-APY-\{3,5-ClSal\})}$$

$$R_2TeCl_2 + \text{(2-APY-\{3,5-ClSalH\})} \longrightarrow R_2TeCl\text{(2-APY-\{3,5-ClSal\})}$$

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 organyltellurium(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 13.22-49.77 S cm^2 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 $TeCl_2$(2-APY-{3,5-ClSal})$^+$ / RTeCl (2-APY-{3,5-ClSal})$^+$ / R_2Te(2-APY-{3,5-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 2-APY-{3, 5-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 IR spectra for our studied complexes give information about the coordination of ligand to metal (Table 2). The IR spectra of Schiff base shows a strong band at 1610 cm^{-1} confirming the formation of azomethine linkage 37, 65, 69. A comparison of the spectra of the tellurium complexes and Schiff base indicates that in all the complexes tellurium atom is coordinated to the Schiff base through the imino nitrogen(s) due to the downfield shift [2, 13, 65, 69-73] in the stretching band of the C=N group on going from ligand (1610 cm^{-1}) to complexes (1471-1489 cm^{-1})

The broad band at 3055 cm^{-1} in free ligand disappears in the complexes, thereby exhibiting the coordination of ligand to the metal through phenolic -OH group via deprotonation. Schiff base

Table 1: Analytical Data, Molar Conductance and Physical Properties for Schiff Base (2-APY-{3, 5-ClSalH}) Complexes of Tellurium (IV)

Compound No.	**Complex (R)**	**Empirical Formula (Formula Wt.)**	**Colour (Yiel, %)**	**M. Pt. (°C) dec.**	**Analyses % Found (Calculated)**					**Λ_M at *ca.* 10^{-3}M S cm^2mol^{-1} in DMSO**
					C	**H**	**N**	**Te**	**Cl**	
Schiff Base	(2-APY-{3,5-ClSalH})	$C_{12}H_8Cl_2N_2O$ (267.14)	Orange (96)	160-162	53.72 (53.95)	3.21 (2.99)	10.35 (10.49)	-	26.32 (26.58)	-
1	$TeCl_3$(2-APY-{3,5-ClSal})	$C_{12}H_7Cl_5N_2OTe$ (500.24)	Light Yellow (82)	140-142	28.64 (28.81)	1.52 (1.40)	5.76 (5.60)	29.32 (29.58)	35.25 (35.48)	13.22
2	$RTeCl_2$(2-APY-{3,5-ClSal}) (4-methoxyphenyl)	$C_{19}H_{14}Cl_4N_2O_2Te$ (571.81)	Cream (78)	120-122	39.72 (39.91)	2.57 (2.45)	4.72 (4.90)	25.17 (25.37)	24.58 (24.83)	19.23
3	$RTeCl_2$(2-APY-{3,5-ClSal}) (4-hydroxyphenyl)	$C_{18}H_{12}Cl_4N_2O_2Te$ (557.80)	Pale Yellow (72)	222-224	38.54 (38.76)	2.03 (2.15)	5.24 (5.02)	26.24 (26.10)	25.58 (25.46)	26.39
4	$RTeCl_2$(2-APY-{3,5-ClSal}) (3-methyl-4-hydroxyphenyl)	$C_{19}H_{14}Cl_4N_2O_2Te$ (571.81)	Light Brown (75)	204-206	39.75 (39.91)	2.28 (2.45)	5.09 (4.90)	25.54 (25.37)	24.58 (24.83)	21.10
5	R_2TeCl(2-APY-{3,5-ClSal})	$C_{26}H_{21}Cl_3N_2O_3Te$	Light	228-230	48.28	3.43	4.23	22.05	16.35	34.80

Compound No.	Complex (R)	Empirical Formula (Formula Wt.)	Colour (Yiel, %)	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	
	(4-methoxyphenyl)	(643.38)	orange (79)		(48.53)	(3.26)	(4.36)	(22.21)	(16.55)	
6	R_2TeCl(2-APY-{3,5-ClSal}) (4-hydroxyphenyl)	$C_{24}H_{17}Cl_3N_2O_3Te$ (615.36)	Dark brown (83)	168-170	46.54 (46.84)	2.52 (2.76)	4.27 (4.55)	23.17 (23.35)	17.18 (17.31)	49.77
7	R_2TeCl(2-APY-{3,5-ClSal}) (3-methyl-4-hydroxyphenyl)	$C_{26}H_{21}Cl_3N_2O_3Te$ (643.38)	Brown (84)	212-214	48.25 (48.53)	3.12 (3.26)	4.49 (4.36)	22.42 (22.21)	16.35 (16.55)	42.34

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

coordination through pyridine nitrogen affecting the $\upsilon_{(C\text{-}N\text{-}C)}$ band, therefore the frequency in free ligand at 1461 cm^{-1} was moved to a higher value and appears in the region 1471-1489 cm^{-1} in the spectrum of complexes.

The bands for $\upsilon_{(Te\text{-}O)}$ mode [74, 76] appeared in the range of 286-291 cm^{-1} and for $\upsilon_{(Te\text{-}N)}$ mode 76 the bands in the region 415-419 cm^{-1} further support the involvement of phenolic oxygen and azomethine nitrogen atom of Schiff base in the coordination.

Further, presence of organyl groups of RTe and R_2Te 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 a hexacoordinated chelating rings with tellurium centre.

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

Compound No.	(Phenolic) $\nu_{(OH)}$	(Azomethine) $\nu_{(C=N)}$	(Pyridine) $\nu_{(C\text{-}N\text{-}C)}$	$\nu_{(Te\text{-}O)}$	$\nu_{(Te\text{-}N)}$
(2-APY-{3,5-ClSalH})	3055 b	1610 s	1461 s	-	-
1	-	1589 s	1471 s	289 s	419 s
2	-	1590 sh	1488 s	290 s	419 s
3	-	1593 s	1486 mb	290 s	419 s
4	-	1590 s	1486 s	286 s	415 b
5	-	1585 sh	1477 s	289 s	419 s
6	-	1592 s	1486 s	291 s	419 s
7	-	1575 s	1489 s	289 s	418 s

(s = strong, m = medium, b = broad, sh = shoulder) *^{1}H NMR Spectra*

The ^{1}H NMR spectral data for free ligand and its complexes are presented in Table 3.

The ligand shows two singlets at 14.539 δ ppm and 9.422 δ ppm corresponding to phenolic –OH, azomethine proton –N=CH- respectively, one complex multiplets at 7.262-8.541 δ ppm corresponding to aromatic as well as pyridine protons.

The presence of –N=CH- proton signal at 9.422 δ ppm in the ligand confirms the formation of base by condensation of 3, 5-dichlorosalicylaldehyde and 2-aminopyridine Schiff base.

Upon complexation the azomethine proton signal in the tellurium (IV) complexes are shifted to downfield side suggesting deshielding of azomethine proton due to cooedination to tellurium through the azomethine nitrogen [48, 49, 69, 77].

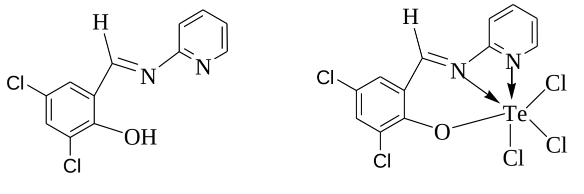
The peak at 14.539 δ ppm in case of ligand disappears in the complexes indicating the involvement of phenolic oxygen of 3, 5-dichlorosalicylaldehyde in the coordination via deprotonation.

Table 3: ^{1}H NMR Spectral Data of Schiff Base (2-APY-{3, 5-ClSalH}) and Complexes in DMSO-d_6.

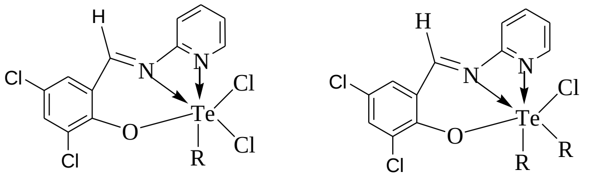
Compound Number	(Phenolic)-OH δ ppm	(Azomethine) -HC=N δ ppm	(Ar rings protons) δ ppm	-CH_3/-OCH_3* δ ppm	-OH of RTe/ R_2Te δ ppm
(2-APY- {3,5-ClSalH})	14.539 (s, 1H)	9.422 (s, 1H)	7.262-8.541 (cm, 6H)	-	-
1	-	10.232 (s, 1H)	7.064-8.016 (cm, 6H)	-	-
2	-	10.236 (s, 1H)	7.039-8.389 (cm, 10H)	3.810 (s,3H*)	-
3	-	10.274 (s, 1H)	6.853-8.103 (cm, 10H)	-	10.005 (s,1H)
4	-	10.235 (s, 1H)	6.840-8.238 (cm, 9H)	2.508 (s,3H)	8.989 (s,1H)
5	-	10.239 (s, 1H)	7.534-8.017 (cm, 14H)	3.812 (s,6H*)	-
6	-	10.235 (s, 1H)	6.838-8.238 (cm, 14H)	-	8.989 (s,2H)

s = singlet, cm = complex multiplet Spectra of compound number 7 not well resolved due to poor solubility

On the basis of spectral evidences tellurium appears to be hex coordinated in these complexes, presumably in a distorted octahedral environment and proposed structures are shown in Figure 1.



(2-APY-{3,5-ClSalH}) TeCl$_3$(2-APY-{3,5-ClSal})



RTeCl$_2$(2-APY-{3,5-ClSal}) R$_2$TeCl(2-APY-{3,5-ClSal})

Fig 1: Proposed Structures of Schiff base and Tellurium (IV) Complexes

Biological Studies

The 3,5-dichlorosalicylaldehyde-2-aminopyridine Schiff base and newly synthesized organyltellurium (IV) Schiff base complexes were evaluated 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 show the complexes of organyltellurium (IV) exhibit more antibacterial activity than antifungal activity as compared to free ligand. The complex no. 2 i.e. [RTeCl$_2$(2-APY-{3,5-ClSal})]: where R = 4-methoxyphenyl, shows good activity against *S. pyogenes* bacterial strain than Schiff base itself and all complexes. Most of the complexes are moderately more effective against bacterial strain *S. aureus* MTCC 96 and *S. pyogenes* MTCC 442 as compare to free ligand.

Table 4: Minimum Inhibitory Concentration MIC (µg/mL) of Schiff Base (2-APY-{3,5-ClSalH}) and Complexes

Compound Number	Bacterial Strain				Fungal Strain		
	***S. aureus* MTCC 96**	***S. pyogenes* MTCC 442**	***P. aeruginosa* MTCC 1688**	***E. coli* MTCC 443**	***C. albicans* MTCC 227**	***A. niger* MTCC 282**	***A. clavatus* MTCC 1323**
(2-APY-{3,5-ClSalH})	500	250	200	125	250	1000	1000
1	200	100	200	200	500	1000	1000
2	100	62.5	250	250	1000	500	1000
3	100	125	500	500	1000	> 1000	> 1000
6	200	250	200	125	500	1000	> 1000
Standard Drugs							
Ampicillin	250	100	100	100	-	-	-
Chloramphenicol	50	50	50	50	-	-	-
Nystatin	-	-	-	-	100	100	100
Greseofulvin	-	-	-	-	500	100	100

4. Conclusions

The Schiff base (2-APY-{3,5-ClSalH}) has been prepared by condensation of 3,5-dichlorosalicylaldehyde with 2-aminopyridine Schiff base when reacted with tellurium tetrachloride, organyltellurium (IV) trichlorides and diorganyltellurium (IV) dichlorides in 1:1 molar ratios yield **[**TeCl$_3$(2-APY-{3,5-ClSal})], [RTeCl$_2$(2-APY-{3,5-ClSal})] and [R$_2$TeCl(2-APY-{3,5-ClSal})]: where R = 4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl type complexes. Spectral studies predict the hexacoordinated tellurium centre by the monobasic tridentate (*ONN*) Schiff base. Some of these complexes possess substantial antimicrobial activity.

5. Acknowledgements

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 SAIF, Panjab University Chandigarh for providing the CHN analyses, ^{1}H NMR spectral data from CIL, Guru Jambeshwar University of Science and Technology, Hissar and the Microcare Laboratory, Surat, for providing antimicrobial activities.

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