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Studies on Some Tellurium(IV) Complexes of Salicylaldehyde-2-Aminopyridine Schiff Base.

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ABSTRACT

The reaction of tellurium(IV) complexes, $TeCl_4$, $ArTeCl_3$ and Ar_2TeCl_2 with tridentate Schiff base of salicylaldehyde with 2-aminopyridine have been carried out. The complexes were characterized by elemental analyses, conductance measurement, FT-IR and proton NMR spectral studies and formulated as **[**$TeCl_3$(2-APY-{Sal})], [$ArTeCl_2$(2-APY-{Sal})] and [Ar_2TeCl(2-APY-{Sal})]: where Ar = p-methoxyphenyl, p-hydroxyphenyl, 3-methyl-4-hydroxyphenyl. The hexacoordinated tellurium (IV) complexes probably in a distorted octahedral geometry have been tentatively proposed for the new complexes. The ligand (2-APY-{SalH}) and some of the metal complexes have been screened for their antifungal and antibacterial activities.

Keywords: Salicylaldehyde, 2-aminopyridine, Tridentate, Antibacterial and Antifungal.

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INTRODUCTION

Compounds with the structure of AC=NB are known as Schiff bases [1], which are easily synthesized from the condensation of primary amines and active carbonyl groups. Some Schiff bases are reported to possess antibacterial [1-7], antifungal [4-8] and antitumor activities [9-10]. Some researchers [11-14] have studied the structure – activity relationship (SAR) as well. Schiff bases are of paramount importance as ligands in metal – coordination chemistry as they form stable complexes with most of transition metals and are also known to stabilize them in various oxidation states [15-20]. It is reported [1, 21] in the literature that heteroatom increases the Schiff base activity and also Schiff bases derived from substituted salicylaldehyde exhibit more antimicrobial activity [22, 23].

Also, aryltellurium(IV) trichlorides are known [24-43] to behave as lewis acids and form complexes with several N- , O- and S- donor bases. The, diaryltellurium(IV) dichlorides are also reported to act as acceptors but much weaker than aryltellurium(IV) trichlorides [38-46]. In view of this, we have investigated the reactions of tellurium(IV) chloride, some aryltellurium trichlorides, $ArTeCl_3$ and diaryltellurium dichlorides, Ar_2TeCl_2 with salicylaldehyde-2-aminopyridine Schiff base (2-APY-{SalH}), to synthesize some new complexes of tellurium(IV).

MATERIALS AND METHODS

All chemicals used were of Analytical Reagent grade. All preparations were carried out under an atmosphere of dry nitrogen as the compounds are sensitive to moisture. The solvents were purified by standard method [47, 48] before use. The purity of compounds was checked by TLC using Silica gel-G (Himedia). 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 Aryltellurium(IV) Trichlorides and Diaryltellurium(IV) Dichlorides

p-Methoxyphenyltellurium(IV) trichloride [49, 50], bis(p-methoxyphenyl)tellurium(IV) dichloride [50, 51], p-hydroxyphenyltellurium(IV) trichloride [52], bis(p-hydroxyphenyl)tellurium(IV) dichloride [52], **3-methyl-4-hydroxyphenyl**tellurium(IV) trichloride [53] and bis(**3-methyl-4-hydroxypheny**l)tellurium(IV) dichloride [53] 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 [49-53].

Preparation of Salicylaldehyde-2-Aminopyridine Schiff Base (2-APY-{SalH})

The Schiff base has been prepared by mixing equimolecular quantity of salicylaldehyde (0.08 mole, 9.76g) and 2-aminopyridine (0.08 mole, 7.52g) in 10 ml methanol in a round bottomed flask equipped with a condenser [15, 54]. 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 calcium chloride and recrystallized from methanol, a sharp yellow crystalline product was obtained.

Preparation of Complexes

Tellurium tetrachloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides, when reacted with Schiff Base (2-APY-{SalH}) form solid complexes as described below:

$[TeCl_3$(2-APY-{Sal})], $[ArTeCl_2$(2-APY-{Sal})] and $[Ar_2TeCl$(2-APY-{Sal})]

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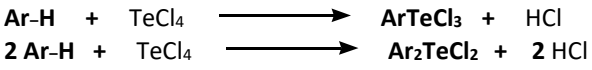
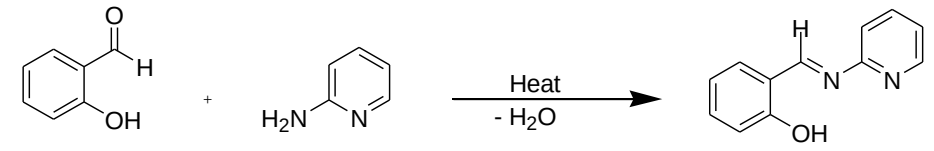
The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL anhydrous methanol to a hot solution of 5 mmol Schiff base in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent was distilled off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

RESULTS AND DISCUSSION

Tellurium tetrachloride when heated with anisole [49-51], phenol [52] **and** o-cresol [53] **(Ar-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 **aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides.**

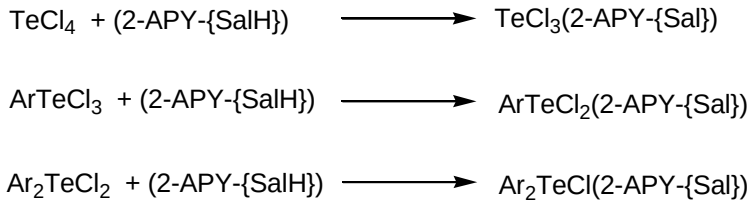


Preparation of Schiff Base (2-APY-{SalH}), by the reaction of salicylaldehyde with 2-aminopyridine can be represented by following equation.



salicylaldehyde 2-aminopyridine (2-APY-{SalH})

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: Analytical Data, Molar Conductance and Physical Properties for Schiff Base (2-APY-{SalH}) 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^2mol^{-1} in DMSO
					C	H	N	Te	Cl	
Schiff Base	(2-APY-{SalH})	$C_{12}H_{10}N_2O$ (198.24)	Yellow (93)	58-60	72.52 (72.70)	5.21 (5.09)	13.96 (14.13)	-	-	-
1	$TeCl_3$(2-APY-{Sal})	$C_{12}H_9Cl_3N_2OTe$ (431.33)	Light purple (79)	211-213	34.69 (34.87)	2.30 (2.11)	6.38 (6.50)	29.72 (29.58)	24.52 (24.69)	16.09
2	$ArTeCl_2$(2-APY-{Sal}) (p-	$C_{19}H_{16}Cl_2N_2O_2Te$ (502.97)	Light-cream (70)	196-198	45.18 (45.37)	3.42 (3.21)	5.25 (5.57)	25.21 (25.37)	13.93 (14.12)	12.86

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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^2mol^{-1} in DMSO
					C	H	N	Te	Cl	
	methoxy phenyl)									
3	$ArTeCl_2$(2-APY-{Sal}) (p-hydroxyphenyl)	$C_{18}H_{14}Cl_2N_2O_2Te$ (488.94)	Brick red (82)	162-164	43.92 (44.21)	2.95 (2.89)	5.62 (5.73)	25.94 (26.10)	14.35 (14.52)	27.11
4	$ArTeCl_2$(2-APY-{Sal}) (3-methyl-4-hydroxyphenyl)	$C_{19}H_{16}Cl_2N_2O_2Te$ (502.97)	Reddish brown (80)	178-180	45.18 (45.37)	3.12 (3.21)	5.43 (5.57)	25.23 (25.37)	14.03 (14.12)	10.16
5	Ar_2TeCl(2-APY-{Sal}) (p-methoxy phenyl)	$C_{26}H_{23}ClN_2O_3Te$ (574.61)	Light pink (78)	186-188	54.17 (54.34)	4.15 (4.04)	4.65 (4.88)	22.42 (22.21)	6.07 (6.18)	22.93
6	Ar_2TeCl(2-APY-{Sal}) (p-hydroxyphenyl)	$C_{24}H_{19}ClN_2O_3Te$ (546.55)	Light brown (68)	169-171	52.43 (52.74)	3.62 (3.51)	5.02 (5.13)	23.47 (23.35)	6.25 (6.50)	39.21
7	Ar_2TeCl(2-APY-{Sal}) (3-methyl-4-hydroxyphenyl)	$C_{26}H_{23}ClN_2O_3Te$ (574.61)	Light- brown (71)	181-183	54.12 (54.34)	4.16 (4.04)	4.59 (4.88)	22.37 (22.21)	6.10 (6.18)	48.30

Values of Λ_M reported [55, 56] for 1:1 electrolytes in DMSO = 50 – 70 S cm^2 mol^{-1}

Conductance Studies

Molar conductance (Λ_M) data for aryltellurium(IV) Schiff base complexes in DMSO are compiled in **Table1**. The Λ_M values at ca. 10^{-3} M of complexes lies in the range 10.16-48.30 S cm^2 mol^{-1} which predict the non-electrolyte to 1:1 weak electrolyte type behavior [55,56] of these Schiff base complexes in DMSO, probably due to ionization into $TeCl_2$(2-APY-{Sal})$^+$ / ArTeCl(2-APY-{Sal})$^+$ / Ar_2Te(2-APY-{Sal})$^+$ 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-{Sal}$^-$ along with Cl^- in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes [57] which are reported to be non-electrolytes.

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Infrared Spectra

The Infrared data of tellurium(IV) complexes and its ligand (2-APY-{SalH}) are presented in **Table2**. The spectra of complexes are quite complex and thus, an attempt has been made to identify the donor sites by comparing the spectra of complexes with parent ligand and $ArTeCl_3/Ar_2TeCl_2$.

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

Compound No.	(Phenolic) $\nu_{(OH)}$	(Azomethine) $\nu_{(C=N)}$	(Pyridine) $\nu_{(C-N-C)}$	$\nu_{(Te-O)}$	$\nu_{(Te-N)}$
(2-APY-{SalH})	3052 b	1607 s	1496 s	-	-
1	-	1580 s	1541 s	294 s	409 s
2	-	1578 s	1542 m	289 s	418 s
3	-	1576 s	1542 s	289 s	419 s
4	-	1577 sh	1541 sh	289 s	418 s
5	-	1578 sh	1544 s	294 s	413 s
6	-	1576 s	1544 m	290 s	419 s
7	-	1576 s	1540 s	294 s	415 s

s = strong, m = medium, b = broad, sh = shoulder

The IR spectrum of the ligand shows three main vibrational bands at 3052, 1607 and 1496 cm^{-1}, which may be assigned [8, 15, 21, 54] to phenolic $\upsilon_{(OH)}$, azomethine $\upsilon_{(C=N)}$ and pyridine $\upsilon_{(C-N-C)}$ group respectively.

In the case of $TeCl_3$(2-APY-{Sal}), $ArTeCl_2$(2-APY-{Sal}) and Ar_2TeCl(2-APY-{Sal}) complexes, $\upsilon_{(C=N)}$ of free (2-APY-{SalH}) at 1607 cm^{-1} upon chelation [8, 15, 54] to metal through azomethine nitrogen atom, shifted to lower wavenumber region between 1592-1576 cm^{-1}. The broad band appearing around 3052 cm^{-1} in free ligand, assignable to $\upsilon_{(OH)}$ vibration mode, is absent in the spectra of complexes suggesting the coordination through oxygen phenolic group (C-O) to the tellurium centre. The another strong absorption band in (2-APY-{SalH}) due to pyridine ring $\upsilon_{(C-N-C)}$ occurring at 1496 cm^{-1} has shifted to higher wavenumber i.e. 1544-1540 cm^{-1} in complexes reflecting that ligand coordinate through nitrogen atom of pyridine ring.

The appearance of new strong bands around at 289- 294 cm^{-1} due to $\upsilon_{(Te-O)}$ [58-61] and in the range of 409-419 cm^{-1} due to $\upsilon_{(Te-N)}$ [62] further support the involvement of phenolic oxygen, azomethine and pyridine nitrogen atoms of Schiff base in the coordination. Further, presence of aryl groups of ArTe and Ar_2Te may result in mixing of certain bands, thus making independent assignments very difficult. Thus, the (2-APY-{SalH}) 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.

^{1}H NMR Spectra

Proton magnetic resonance spectra of aryltellurium(IV) salicylaldehyde-2-aminopyridine complexes are very complex and a lot of mixing of aryl proton signals of the (2-APY-{SalH}) and aryltellurium(IV) moiety takes place, thus making the independent assignment almost impossible. The chemical shift data for the free (2-APY-{SalH}) [15] and their complexes are compiled in **Table3**.

Table 3: ^{1}H NMR Spectral Data of Schiff Base (2-APY-{SalH}) and Complexes.

Compound Number	Chemical Shift, δ ppm in DMSO-d_6
(2-APY-{SalH})	6.933-8.522 (cm, 8H, aromatic protons), **9.445 (s, 1H, -HC=N, azomethine proton), 13.453 (s, 1H, OH)**
1	6.843-8.051(cm, 8H, aromatic protons),

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	10.394 (s, 1H, -HC=N, azomethine proton)
2	3.813 (s, 3H, -OCH_3), 6.831-8.345 (cm, 12H, aromatic protons), **10.316 (s, 1H, -HC=N, azomethine proton)**
3	7.625-8.094 (cm, 12H, aromatic protons), 9.088 (s,1H,phenolic OH of ArTe), **10.139 (s, 1H, -HC=N, azomethine proton),**
5	3.401 (s, 6H, -OCH_3), 6.826-7.953 (cm, 16H, aromatic protons), **10.215 (s, 1H, -HC=N, azomethine proton)**
6	6.962-7.948 (cm, 16H, aromatic protons), **9.908 (s, 1H, -HC=N, azomethine proton),** 8.015 (s, 2H, phenolic OH of Ar_2Te)
7	2.499 (s, 6H, -CH_3), 6.853-7.946 (cm, 14H, aromatic protons), **10.213 (s, 1H, -HC=N, azomethine proton),** 8.213 (bs, 2H, phenolic OH of Ar_2Te)

s = singlet, cm = complex multiplet, bs = broad singlet.

Free ligand shows three signals at 13.453 (singlet), 9.445 (singlet) and 6.933-8.522 (complex multiplet) δ ppm due phenolic OH of salicylaldehyde, azomethine proton and aromatic ring as well as pyridine protons respectively.

In the proton NMR of complexes i.e. $TeCl_3$(2-APY-{Sal}), $ArTeCl_2$(2-APY-{Sal}) and Ar_2TeCl(2-APY-{Sal}) the singlet at 13.453 δ ppm, in free ligand, disappear on complexation indicating the involvement of phenolic oxygen in coordination via deprotonation. Another signal displays a downfield shift from 9.445 to 9.908-10.394 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 [15, 32, 33]. Independent assignments to the aryl protons of (2-APY-{SalH}) and ArTe / Ar_2Te are not possible due to overlapping of signals in this region.

On the basis of spectral studies, it may be concluded that (2-APY-{SalH}) 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 **Figure1**.

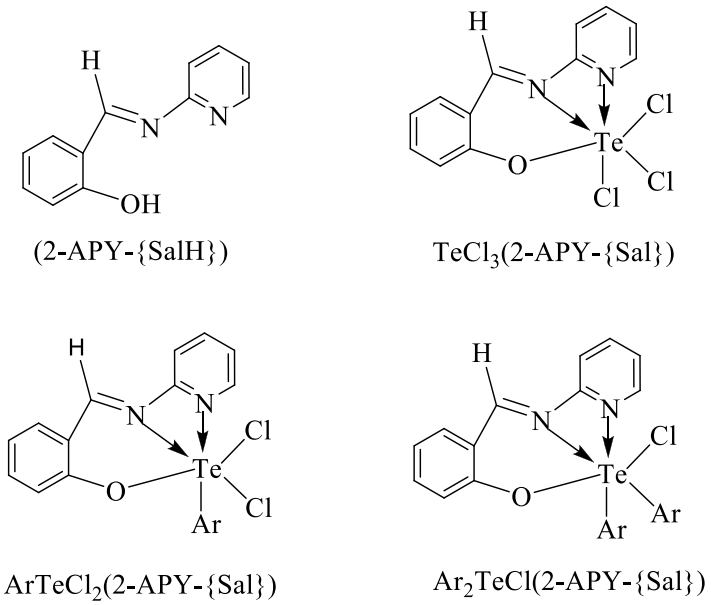

Figure 1: Proposed Structures of Schiff base (2-APY-{SalH}) and Tellurium (IV) Complexes

Biological Studies

Anti bacterial and antifungal activities of the ligand (2-APY-{SalH}) and its newly synthesized complexes were carried out 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 respectively. The evaluation of the biological activities was carried by "Broth Dilution Method". The lowest concentration inhibiting growth of the organism is recorded as the MIC. The MIC values of ligand and some complexes have been compared with standard drugs ampicillin and chloramphenicol for antibacterial, nystatin and greseofulvin for antifungal activities [63, 64] and are given in **Table4**.

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

Compound Number	Bacterial Strain				Fungal Strain		
	S. Aureus MTCC96	S. pyogenes MTCC442	P. aeruginosa MTCC1688	E. coli MTCC443	C. albicans MTCC227	A. Niger MTCC282	A. clavatus MTCC1323
(2-APY-{SalH})	500	500	500	500	>1000	200	250
1	200	200	250	125	500	500	1000
2	250	200	250	200	500	250	250
3	125	125	250	500	1000	250	250
6	200	200	250	200	1000	>1000	>1000
Standard Drugs							
Ampicillin	250	100	100	100	-	-	-
Chloramphenicol	50	50	50	50	-	-	-
Nystatin	-	-	-	-	100	100	100
Greseofulvin	-	-	-	-	500	100	100

The data reveal that the new tellurim(IV) complexes possess substantial antimicrobial activity especially against bacteria than Schiff base itself. It has been observed that complex 3 possess more antibacterial activity against S. aureus even than the standard drug ampicillin.

CONCLUSION

The new complexes of tellurium(IV) derivatives upon reaction with Schiff base (2-APY-{SalH}) derived from salicylaldehyde and 2-aminopyridine are reported. These newly synthesized complexes i.e. $TeCl_3$(2-APY-{Sal}), $ArTeCl_2$(2-APY-{Sal}) and Ar_2TeCl(2-APY-{Sal}) were characterized by elemental analyses, conductance measurement, IR and proton NMR spectral studies. The hexacoordinated tellurium(IV) complexes probably in a distorted octahedral geometry have been tentatively proposed for these new complexes. Based on these studies, salicylaldehyde-2-aminopyridine Schiff Base ligand behaves as uninegative tridentates (ONN) ligand. The complexes have been observed to exhibit more antimicrobial activity towards bacteria as compared to fungii.

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