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Investigations on Some Isatin- p-Toluidine Schiff Base Complexes of Tellurium (IV)

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ABSTRACT

Fourteen new tellurium (IV) complexes with Schiff base of isatin (1-H-indol-2,3-dione) with p-toluidine have been synthesized under different experimental conditions to yield two types of complexes: $\text{TeCl}_4(\text{HL})$, $\text{RTeCl}_3(\text{HL})$, $\text{R}_2\text{TeCl}_2(\text{HL})$ and $\text{TeCl}_2(\text{L})_2$, $\text{RTeCl}(\text{L})_2$, $\text{R}_2\text{Te}(\text{L})_2$; where R = p-methoxyphenyl, p-hydroxyphenyl, 3-methyl-4-hydroxyphenyl and HL = HIPT, isatin- p-toluidine Schiff base. These complexes have been characterized by elemental analyses, conductance measurement, infrared and proton magnetic resonance spectroscopy. The spectral studies predict that Schiff base acts as a neutral bidentate (N,O) and monobasic uninegative bidentate (N,O) donor giving hexacoordinated tellurium(IV) complexes probably in a distorted octahedral environment. Some of these complexes have also been observed to possess biocidal activity against bacteria and fungus.

Keywords: Isatin- p-toluidine Schiff base, Tellurium tetrachloride, Aryltellurium(IV), Diaryltellurium(IV) and Biocidal activity.

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INTRODUCTION

Isatin (1-H-indol-2,3-dione) and its derivatives are reported to exhibit a range of promising pharmacodynamic profile like anticonvulsant [1,2], anti-HIV [3], cytotoxic [4], tuberculostatic [5], and antimicrobial [6]. A large number of Schiff's bases of isatin have been reported in the literature which undergoes complexation with various metal ions in different modes [7-15]. Schiff bases derived from isatin with p-toluidine can act as a ligand [7, 14] having functional groups with nitrogen and oxygen donor atoms.

Also, aryltellurium(IV) trichlorides are known [16-29] to behave as lewis acids and form complexes with several nitrogen, oxygen and sulphur donor bases. The, diaryltellurium(IV) dichlorides are also reported to act as acceptors but much weaker than corresponding aryltellurium(IV) trichlorides [30-32]. Tellurium(IV) chloride is also known to form adducts with nitrogen and sulphur donors [33-35], thus reflecting its acceptor behaviour. In view of this and in continuation of earlier work on isatin Schiff bases [36] we herein report the studies on reactions of tellurium(IV) chloride, aryltellurium trichlorides, RTeCl_3 and diaryltellurium dichlorides, R_2TeCl_2 with Isatin- p-toluidine Schiff base (HIPT), to synthesize some new complexes of tellurium(IV).

MATERIALS AND METHODS

All preparations were carried out under an atmosphere of dry N_2 as the compounds are sensitive to moisture. The solvents were dried by standard method before use. The purity of compounds was checked by thin layer chromatography 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. Conductance measurements were carried out in DMSO at $25 \pm 2^\circ\text{C}$ with a dip type conductivity cell (cell constant = 1.017) on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra ($4000\text{-}400\text{ cm}^{-1}$) were recorded in KBr pellets at SAIF, Panjab University Chandigarh on a F.T. Infra-Red Spectrophotometer Model RZX (Perkin Elmer). Proton NMR Spectra were recorded in DMSO-d_6 using TMS as an internal reference on BRUKER AVANCE II 400 NMR spectrometer.

Biological activity of some of the complexes was evaluated by determination of zone of inhibition. Zone of inhibition of Schiff base and some complexes has been measured by agar well diffusion assay [37]. The inoculums suspensions of the test microorganisms were prepared by using 16 h old cultures adjusted to 10^8 cfu/mL by referring the 0.5 McFarland standards. Total 20 mL of Nutrient agar medium was poured into each petri plate and then plates were swabbed with 100 μL inoculums of the test microorganisms and kept for 15 min for adsorption. Wells were bored into the seeded agar plates using a sterile cork borer of diameter (8 mm), and these were loaded with a 20 μL volume with concentration of 10 mg/mL of each compound reconstituted in the DMSO. The incubation of all the plates was carried at 37°C for 24 hours. Antimicrobial activity against the selected organisms was evaluated by measuring the zone of inhibition with zone reader (Hi antibiotic zone scale).

Preparation of Aryltellurium(IV) Trichlorides, RTeCl_3 and Diaryltellurium(IV) Dichlorides, R_2TeCl_2

p-Methoxyphenyltellurium(IV) trichloride [38, 39], bis(p-methoxyphenyl)tellurium(IV) dichloride [39, 40], p-hydroxyphenyltellurium(IV) trichloride [41], bis(p-hydroxyphenyl)tellurium(IV) dichloride [41], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride [42] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride [42] were prepared by the reactions of TeCl_4 with anisole /phenol /o-cresol as reported in the literature [38-42].

Preparation of Isatin - p-Toluidine Schiff Base (HIPT)

The Schiff base was prepared by method reported by Kriza and Parnau [14]. Saturated solutions of equimolar quantities of isatin and p-toluidine in dry ethanol were refluxed for 3 hours. After completion of reaction, the reaction mixture was kept overnight to get the orange coloured solid product. The product was filtered, dried and recrystallized from ethanol.

Yield = 92%, M. Pt. = 166-168 °C.

Analyses (calculated %) C₁₅H₁₂N₂O: C (76.28), H (5.08) and N (11.86); found: C 75.85, H 5.51 and N 11.52.

Preparation of Complexes

Tellurium tetrachloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides, when reacted with isatin- *p*-toluidine Schiff base (HIPT) form two types of complexes under different experimental conditions as described below:

(a) TeCl₄ (HL), RTeCl₃ (HL) and R₂TeCl₂ (HL)

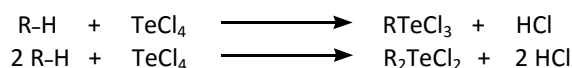
These types of complexes were prepared by addition of hot methanolic solution of the HIPT (5 mmol in about 25 mL) to a solution of tellurium (IV) derivatives (5 mmol) in about 25 mL dry methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent was distilled off to obtain the desired products, which were recrystallized from dry methanol.

(b) TeCl₂ (L)₂, RTeCl (L)₂, R₂Te (L)₂

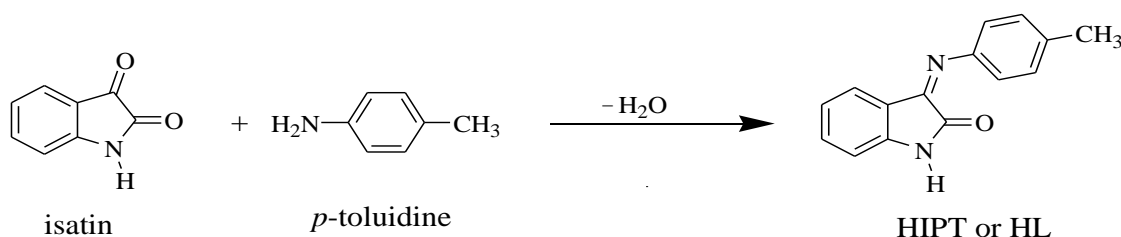
These complexes were prepared by addition of tellurium (IV) compounds i.e. TeCl₄ / RTeCl₃ / R₂TeCl₂ (5 mmol) in 25 ml dry methanol to a hot methanolic solution of the HIPT (10 mmol / 50 mL) with continuous stirring. Sodium methoxide was then added until pH reached to about 7.4. Contents were then stirred for about 1 hour and white turbidity obtained was removed by filtration. The reaction mixture was then refluxed on steam bath for about 6 hours. The excess solvent was distilled off to about one third of original volume and kept at room temperature overnight. The coloured complexes crystallized out, which were filtered, washed with dry methanol and dried in a vacuum desiccator over P₄O₁₀.

RESULTS AND DISCUSSION

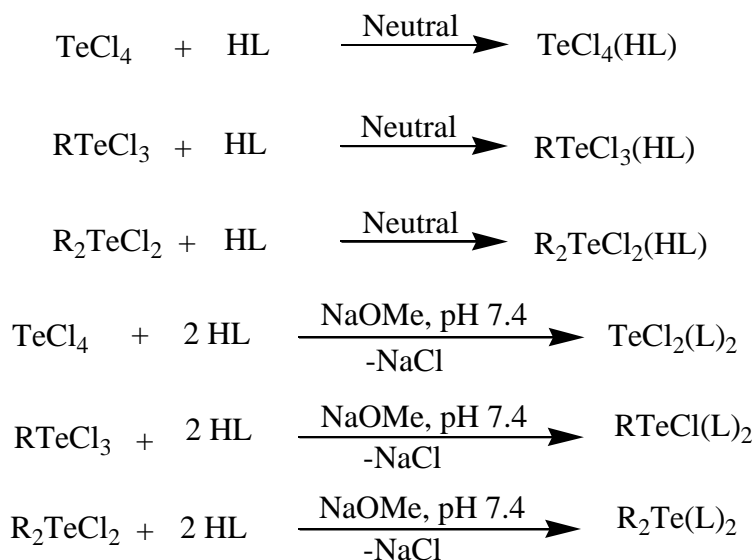
The formation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides by the reactions of TeCl₄ with anisole [38-40], phenol [41] and *o*-cresol [42] (R-H) involves the electrophilic substitution of the aromatic ring by a chlorotellurium group at a position para to the methoxy / hydroxyl group.



Preparation of isatin - *p*-toluidine Schiff base (HIPT), by the reaction of isatin with *p*-toluidine can be represented by following equation:



Isatin - *p*-toluidine Schiff Base (HIPT) reacts with tellurium(IV) chloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides in 1:1 (neutral) and 2:1 (basic) molar ratios to form the corresponding 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 organic solvents, but are soluble in polar donor organic solvents like DMF, DMSO etc. The analytical data and physical properties of Schiff base and the tellurium (IV) complexes are recorded in Table 1.

Conductance Studies

Molar conductance (Λ_M) data for the complexes in DMSO are presented in Table 1. The Λ_M values at ca. 10^{-3} M predict the weak to 1:1 electrolyte [43, 44] type behavior of these Schiff base complexes in DMSO, probably due to ionization into $\text{TeCl}_3(\text{HL})^+ / \text{RTeCl}_2(\text{HL})^+ / \text{R}_2\text{TeCl}(\text{HL})^+ / \text{TeCl}(\text{L})_2^+ / \text{RTe}(\text{L})_2^+$ and Cl^- in solutions of 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 L^- along with Cl^- in DMSO solutions.

This conductance behavior of tellurium (IV) – isatin p-toluidine Schiff base complexes is different from such complexes of Sn(IV) [14], Ti(IV) and Zr(IV) [7], which are observed to be non-electrolytes in dimethylformamide.

Infrared Spectra

The Infrared spectra of HIPT and its tellurium(IV) complexes are presented in Table 2. The spectra of isatin- p-toluidine Schiff base complexes are quite complex and thus an attempt has been made to identify the donor sites by comparing the spectra of complexes with the parent ligand and aryltellurium(IV) chlorides. The IR spectrum of the ligand (HIPT) shows strong bands at 3192 , 1741 and 1651 cm^{-1} , which may be assigned [7, 14] to ν_{NH} , $\nu_{\text{C=O}}$ and $\nu_{\text{C=N}}$ of azomethine group, respectively.

In the case of $\text{TeCl}_4(\text{HL})$, $\text{RTeCl}_3(\text{HL})$ and $\text{R}_2\text{TeCl}_2(\text{HL})$ complexes, $\nu_{(\text{C=O})}$ of free HIPT at 1741 cm^{-1} was shifted to lower wave numbers between 1733 - 1728 cm^{-1} . This shift indicates [7, 14] the implication of carbonyl oxygen in the coordination at tellurium centre. The band appearing at 1651 cm^{-1} in free HIPT, assignable to $\nu_{\text{C=N}}$ vibration mode, disappears in the spectra of complexes and strong band at around 1617 - 1611 cm^{-1} appears indicating participation of azomethinic nitrogen atom in the coordination. Also, the ν_{NH} band in HIPT at 3192 (broad) remains unaffected in the complexes, showing its non participation in bonding with the tellurium atom. Thus, the neutral HL Schiff base is coordinated to the tellurium atom as a NO bidentate ligand.

In the infrared spectra of $\text{TeCl}_2(\text{L})_2$, $\text{RTeCl}(\text{L})_2$ and $\text{R}_2\text{Te}(\text{L})_2$, new IR bands are observed at 1592 - 1573 cm^{-1} and 1277 - 1236 cm^{-1} , which may be assigned to $\nu_{\text{C=N}}^*$ (new azomethine bond) and $\nu_{\text{C-O}}$ vibration modes respectively due to enolization of NH hydrogen and coordination through oxygen of C-O group after deprotonation [7, 14]. All these complexes exhibit strong band at around 1615 cm^{-1} due to coordinated azomethinic nitrogen atoms. Thus, HIPT acts as a uninegative NO bidentate ligand in these 1:2 complexes. The broad band at 3192 cm^{-1} in free ligand disappear in the complexes, there by exhibiting enolization of NH proton.

Table 1: Analytical Data, Molar Conductance and Physical Properties for Isatin- p-Toluidine Schiff Base 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. $10^{-3}M$ $ohm^{-1} cm^2 mol^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	HIPT / HL	$C_{15}H_{12}N_2O$ (236.18)	Orange (92)	166-168	75.85 (76.28)	5.51 (5.08)	11.52 (11.86)	-	-	-
I	$TeCl_4$ (HL)	$C_{15}H_{12}Cl_4N_2OTe$ (505.59)	Brown (90)	154-156	35.28 (35.63)	(2.37)	5.43 (5.54)	25.14 (25.24)	27.79 (28.05)	58.12
II	$TeCl_2$ (L) ₂	$C_{30}H_{22}Cl_2N_4O_2Te$ (668.86)	Light orange (76)	148-150	53.68 (53.87)	3.48 (3.29)	8.21 (8.38)	18.82 (19.08)	10.42 (10.60)	72.61
III	$RTeCl_3$ (HL) (p-methoxyphenyl)	$C_{22}H_{19}Cl_3N_2O_2Te$ (577.21)	Yellow (81)	181-183	45.27 (45.78)	3.57 (3.29)	4.62 (4.85)	21.94 (22.11)	18.21 (18.43)	42.19
IV	$RTeCl(L)_2$ (p-methoxyphenyl)	$C_{37}H_{29}ClN_4O_3Te$ (740.48)	Yellow orange (85)	186-188	59.80 (60.02)	4.17 (3.92)	7.14 (7.57)	16.95 (17.23)	4.56 (4.79)	51.56
V	$RTeCl_3$ (HL) (p-hydroxyphenyl)	$C_{21}H_{17}Cl_3N_2O_2Te$ (563.20)	Light brown (78)	142-144	44.37 (44.79)	3.21 (3.02)	4.72 (4.97)	22.47 (22.66)	18.62 (18.88)	49.73
VI	$RTeCl(L)_2$ (p-hydroxyphenyl)	$C_{36}H_{27}ClN_4O_3Te$ (726.47)	Yellow (82)	220-222	59.25 (59.52)	3.94 (3.72)	7.45 (7.71)	17.32 (17.56)	4.72 (4.88)	62.44
VII	$RTeCl_3$ (HL) (3-methyl-4-hydroxyphenyl)	$C_{22}H_{19}Cl_3N_2O_2Te$ (577.21)	Light brown (82)	160-162	44.45 (45.78)	3.47 (3.29)	4.55 (4.85)	21.94 (22.11)	18.21 (18.43)	53.49
VIII	$RTeCl(L)_2$ (3-methyl-4-hydroxyphenyl)	$C_{37}H_{29}ClN_4O_3Te$ (740.48)	Dark yellow (90)	154-156	59.75 (60.02)	4.17 (3.92)	7.20 (7.57)	16.95 (17.23)	4.52 (4.79)	44.85
IX	R_2TeCl_2 (HL) (p-methoxyphenyl)	$C_{29}H_{26}Cl_2N_2O_3Te$ (648.84)	Dark yellow (81)	112-114	53.42 (53.68)	4.25 (4.01)	4.12 (4.32)	19.54 (19.67)	10.75 (10.93)	39.36
X	$R_2Te(L)_2$ (p-methoxyphenyl)	$C_{44}H_{36}N_4O_4Te$ (812.11)	Yellow (88)	178-180	64.92 (65.08)	4.62 (4.43)	6.78 (6.90)	15.52 (15.71)	-	41.70
XI	R_2TeCl_2 (HL) (p-hydroxyphenyl)	$C_{27}H_{22}Cl_2N_2O_3Te$ (620.81)	Red (76)	202-204	52.04 (52.24)	3.85 (3.54)	4.34 (4.51)	20.32 (20.55)	11.28 (11.42)	13.88

Compound No.	Complex (R)	Empirical Formula (Formula Wt.)	Colour (Yield, %)	M. Pt. (°C) dec.	Analyses % Found (Calculated)					Λ_M at ca. $10^{-3}M$ $ohm^{-1} cm^2 mol^{-1}$ in DMSO
					C	H	N	Te	Cl	
XII	$R_2Te(L)_2$ (p-hydroxyphenyl)	$C_{42}H_{32}N_4O_4Te$ (784.09)	Orange (84)	116-118	64.23 (64.34)	4.35 (4.08)	7.01 (7.15)	16.05 (16.27)	-	17.72
XIII	$R_2TeCl_2(HL)$ (3-methyl-4-hydroxyphenyl)	$C_{29}H_{26}Cl_2N_2O_3Te$ (648.84)	Reddish brown (69)	211-213	53.35 (53.68)	4.26 (4.01)	4.14 (4.32)	19.46 (19.67)	10.65 (10.93)	18.22
XIV	$R_2Te(L)_2$ (3-methyl-4-hydroxyphenyl)	$C_{44}H_{36}N_4O_4Te$ (812.11)	Light orange (74)	132-134	64.84 (65.08)	4.67 (4.43)	6.75 (6.90)	15.45 (15.71)	-	64.76

Values of Λ_M reported [43, 44] for 1:1 electrolytes in DMSO = 50 – 70 $ohm^{-1} cm^2 mol^{-1}$

Table 2: Important IR Data (cm^{-1}) of Isatin- p-Toluidine Schiff Base Complexes of Tellurium (IV)

Compound No.	ν_{NH}	$\nu_{C=O}$	$\nu_{C=N}$	$\nu_{C=N^*}$	ν_{C-O}
HIPT/ HL	3192 sb	1741 s	1651 s	-	-
I	3189 s	1728 s	1616 s	-	-
II	-	-	1616 s	1582 sh	1277 m
III	3192 mb	1730 s	1613 s	-	-
IV	-	-	1620 sh	1573 s	1246 s
V	3163 mb	1728 s	1616 s	-	-
VI	-	-	1613 s	1584 m, sh	1262 m
VII	3185mb	1731 s	1615 s	-	-
VIII	-	-	1616 sh	1580 s	1252 s
IX	3190 mb	1730 s	1615 s	-	-
X	-	-	1612 s	1585 s	1250 s
XI	3188 s	1733 s	1617 s	-	-
XII	-	-	1611 s	1592 sh	1236 m
XIII	3190 mb	1728 sh	1614 s	-	-
XIV	-	-	1612 s	1589 sh	1254 s

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

Table 3: ¹H NMR Spectral Data of Isatin- p-Toluidine Schiff Base Complexes of Tellurium (IV) in DMSO-d₆.

Compound No	Chemical Shift, δ ppm
HIPT / HL	2.38 (s, 3H, CH ₃), 6.53-6.95 (m, 4H, aryl protons of amine), 7.03-7.57 (m, 4H, aryl protons of isatin), 11.03 (s, 1H, NH)
I	2.35 (s, 3H, CH ₃), 6.55-7.57 (cm, 8H, aryl protons of Schiff base), 11.11 (s, 1H, NH)
III	2.38 (s, 3H, CH ₃), 3.83 (s, 3H, -OCH ₃), 6.54-7.57 (cm, 12H, aryl protons of Schiff base & RTe), 11.08 (s, 1H, NH)
V	2.35 (s, 3H, CH ₃), 6.53-8.26 (cm, 12H, aryl protons of Schiff base & RTe), 9.79 (bs, 1H, phenolic OH of RTe), 11.03 (s, 1H, NH)
VI	2.38 (s, 6H, CH ₃), 6.53-7.57 (cm, 20H, aryl protons of Schiff base & RTe), 10.60 (bs, 1H, phenolic OH of RTe)
VII	2.17, 2.34 (s, 6H, CH ₃), 6.55-8.19 (cm, 11H, aryl protons of Schiff base and RTe), 9.84 (bs, 1H, phenolic OH of RTe), 11.03 (s, 1H, NH)
IX	2.32, 2.38 (s, 3H, CH ₃), 3.83 (s, 6H, -OCH ₃), 6.53-8.38 (cm, 16H, aryl protons of Schiff base and R ₂ Te), 11.06 (s, 1H, NH)
X	2.32, 2.38 (s, 6H, CH ₃), 3.78 (s, 6H, -OCH ₃), 6.53-7.77 (cm, 24H, aryl protons of Schiff base and R ₂ Te)
XII	2.38 (s, 6H, CH ₃), 6.54-7.31 (cm, 24H, aryl protons of Schiff base and R ₂ Te), 10.93 (bs, 2H, phenolic OH of R ₂ Te)
XIII	2.19, 2.38 (s, 9H, CH ₃), 6.53-7.67 (cm, 14H, aryl protons of Schiff base and R ₂ Te), 9.20 (bs, 2H, phenolic OH of R ₂ Te), 11.08 (s, 1H, NH)

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

Table 4: Antimicrobial Study [zone of inhibition (mm)] of Isatin- p-Toluidine Schiff Base Complexes of Tellurium (IV)

Compound Number	Staphylococcus aureus	Bacillus subtilis	Pseudomonas aeruginosa	Escherichia coli	Candida albicans
HIPT / HL	16	12	10	10	5
I	16	12	10	10	5
III	11	10	8	7	3
IV	14	16	14	11	6
V	14	14	12	10	5
VI	10	14	12	9	6
VII	12	11	9	9	4
IX	10	11	9	7	4
XII	14	16	14	11	5
XIV	12	13	11	10	5

Further, presence of aryl groups of RTe and R₂Te may result in mixing of certain bands, thus making independent assignments very difficult. The $\nu_{\text{Te-O}}$ and $\nu_{\text{Te-N}}$ could not be ascertained due to non-availability of Far IR data.

Proton Magnetic Resonance Spectra

¹H NMR spectra of aryltellurium(IV) isatin- p-toluidine complexes are very complex and a lot of mixing of aryl proton signals of the HIPT and aryltellurium(IV) moiety takes place, thus making the independent assignment almost impossible. The chemical shift data for the free HIPT and some of representative isatin- p-toluidine Schiff base complexes are compiled in Table 3.

Free HIPT shows two multiplets at 6.54-6.95 δ ppm and 7.03-7.57 δ ppm corresponding to amine aromatic ring and isatin skeleton, respectively, one singlet at 11.03 δ ppm corresponding to isatin NH residue and one singlet at 2.38 δ ppm due to methyl protons of toluidine ring.

The proton NMR spectra of 1:1 complexes i.e. TeCl₄(HL), RTeCl₃(HL) and R₂TeCl₂(HL) display a downfield shift from 11.03 to 11.11 δ ppm, which is associated with the hydrogen of isatin NH residue. This behavior is related with a decrease of electron density and deshielding of NH proton, as a result of participation of the adjacent carbonyl group in coordination [45, 46]. Independent assignments to the aryl protons of Schiff base and RTe / R₂Te are not possible due to overlapping of signals in this region. Also, absence of NH signals in the ¹H NMR spectra of 1:2 complex, i.e. TeCl₂(L)₂, RTeCl(L)₂ and R₂Te(L)₂ confirms the enolization of NH hydrogen and then coordination at tellurium centre through the oxygen of C-O group, as predicted by infrared spectral studies.

On the basis of spectral studies, it may be concluded that HIPT acts as neutral bidentate (N,O) in 1:1 complexes and as a monobasic bidentate (N,O) ligand in 1:2 type complexes, resulting in the formation of hexacoordinated tellurium(IV) complexes probably in a distorted octahedral geometry as shown in Figure 1.

Biological Studies

Isatin- p-toluidine Schiff base (HIPT) and some of its complexes were evaluated for antibacterial and antifungal activity in vitro against Gram-positive bacteria (Staphylococcus aureus and Bacillus subtilis), Gram-negative bacteria (Pseudomonas aeruginosa and Escherichia coli) and fungus Candida albicans, respectively. Zone of inhibition of Schiff base and its complexes are given in table 4.

The data reveal that the new tellurium (IV) complexes possess substantial antimicrobial activity against bacteria especially towards Bacillus subtilis and Pseudomonas aeruginosa. However, in case of fungus C. albicans, the activity of complexes is comparable to the parent Schiff base.

CONCLUSION

The synthesis and characterization of new complexes of tellurium (IV) derivatives with a Schiff base derived from isatin and p-toluidine are reported. The synthesis were performed under different experimental conditions allowing the neutral bidentate and monobasic bidentate functions of the Schiff base so as to obtain the complexes of TeCl₄(HL), RTeCl₃(HL), R₂TeCl₂(HL); and TeCl₂(L)₂, RTeCl(L)₂, R₂Te(L)₂ types. The new tellurium (IV) complexes were characterized by elemental analyses, conductance measurement, IR and ¹H NMR spectral studies. Based on these studies, a distorted octahedral stereochemistry around central tellurium atom has been assigned to these complexes. The zone of inhibition of some of these complexes has also been evaluated by agar well diffusion assay.

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