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Studies on some propylenediamine-bis(isatin) Schiff base complexes of tellurium(IV)

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ABSTRACT

Some new tellurium(IV) complexes of propylenediamine-bis(Isatin) Schiff base with TeCl_4 , RTeCl_3 and R_2TeCl_2 ; where R = *p*-methoxy-, *p*-hydroxy- and 3-methyl-4-hydroxyphenyl, have been synthesized and characterized via elemental analyses, conductance measurement, infrared and proton magnetic resonance spectral studies. The data suggest 1:1 (tellurium: ligand) stoichiometry of all the complexes where the Schiff base behaves as a neutral ONNO quadridentate ligand. Some of these complexes have also been observed to possess antifungal and antitubercular activity.

Keywords: Propylenediamine, Isatin, Tellurium (IV), Quadridentate, Antimicrobial activity.

INTRODUCTION

Although 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 [1-10], but studies on such Schiff bases derived from isatin and aliphatic diamines are quite few [1,4,6,9,10]. Isatin (Indole-2, 3-dione) and its derivatives are of versatile interest due to their biological and medical applications [11-16].

Also, tellurium (IV) compounds such as TeCl_4 [17-19], aryltellurium(IV) trichlorides [20-33] and diaryltellurium(IV) dichlorides [34-36] are reported to act as acceptors and form complexes with nitrogen, oxygen and sulphur donor bases.

In view of this and in continuation of earlier work on isatin Schiff bases [37, 38], we herein deemed desirable to report the synthesis and characterization of complexes of tellurium(IV) chloride, aryltellurium(IV) trichlorides, RTeCl_3 and diaryltellurium(IV) dichlorides, R_2TeCl_2 with the Schiff base obtained by condensation of isatin with propylenediamine.

MATERIALS AND METHODS

All the chemicals used were of Analytical Reagent Grade. 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.

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 ($4000-400\text{ cm}^{-1}$) 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.

Antifungal activity was evaluated in acetone against *R. solani*, *F. oxysporum* and *C. capsici*. The compounds were prepared in 1000 and 500 ppm concentrations in acetone using Poison Plate Technique method [44]. Potato dextrose-agar (PDA) medium was prepared in flasks and sterilized. To this medium, a requisite quantity of solution was added and then the medium was poured into Petri plates in three replications. A culture of test fungus was grown on PDA for 6–7 days. Small disc (4 mm) of the fungus culture was cut with a sterile cork borer and transferred aseptically, upside-down in the center of Petri dishes containing the medium and fungicides. Plates were incubated at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Colony diameters were measured and data were statistically analysed.

Antitubercular activity was evaluated in DMSO against *M. tuberculosis* H₃₇Rv using Microplate Alamar Blue Assay (MABA) method [45,46]. Antitubercular susceptibility test was performed in black, clear-bottomed, 96-well microplates (Packard Instrument Company, Meriden, Conn., USA) in order to minimize background fluorescence. Primary screening of the compounds for antitubercular activity has been conducted at 12.5 $\mu\text{g/ml}$.

Preparation of Propylenediamine bis-(isatin) Schiff base, [N, N'-Bis(indol -2-oxo-3-ylidene)-1,3-diaminopropane], PDBI or H₂L

The Schiff base was prepared by the method reported by Khalifa and Hassaan [6]. A solution of isatin (0.02 mol) in 50 mL ethanol was added dropwise at the ambient temperature to an ethanolic solution of 0.01 mol propylenediamine with continuous stirring. The contents were refluxed on steam bath for 1 hour and then concentrated *in vacuo* during which time; a reddish brown product was precipitated. The mixture was cooled to room temperature and the precipitate was filtered off, washed with ethanol and then dried in a vacuum desiccator over P₄O₁₀. Yield 90 %, m.pt. 208 - 210 °C.

Preparation of aryltellurium(IV) trichlorides, RTeCl₃ and diaryltellurium(IV) dichlorides, R₂TeCl₂

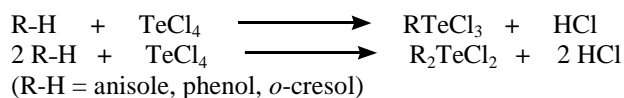
p-Methoxyphenyltellurium(IV) trichloride [39,40], bis(*p*-methoxyphenyl)tellurium(IV) dichloride [40,41], *p*-hydroxyphenyltellurium(IV) trichloride [42], bis(*p*-hydroxyphenyl)tellurium(IV) dichloride [42], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride [43] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride [43] were prepared by the reactions of TeCl₄ with anisole /phenol /*o*-cresol as reported in the literature [39-43].

Preparation of Complexes, TeCl₄ (H₂L), RTeCl₃ (H₂L) and R₂TeCl₂ (H₂L)

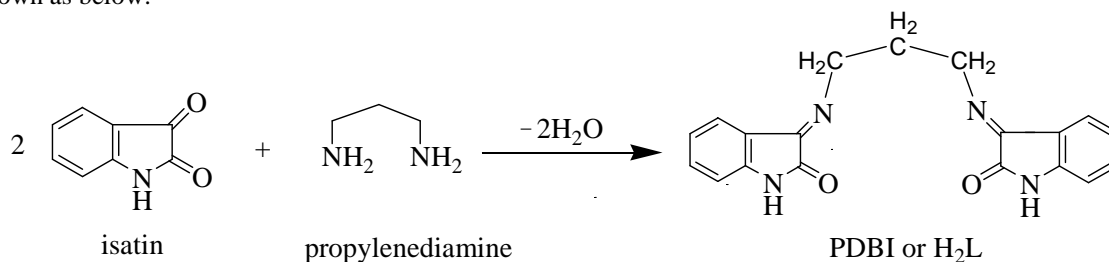
These types of complexes were prepared by addition of hot methanolic solution of the PDBI (10 mmol in about 50 mL) to a solution of tellurium (IV) derivatives (10 mmol) in about 50 mL dry methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent was distilled off to obtain the desired products, which were recrystallized from dry methanol and dried in vacuum desiccator over P₄O₁₀. The reactions were also attempted by addition of sodium methoxide until pH 7.3, but similar products were obtained unlike Sn(IV) and Zr(IV), where Schiff base acts as a binategative tetradentate ligand [1].

RESULTS AND DISCUSSION

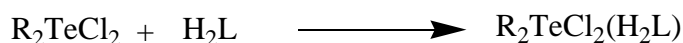
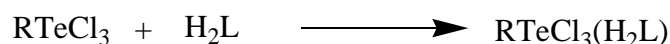
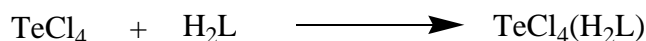
The formation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides by the reactions of TeCl₄ with anisole [39-41], phenol [42] and *o*-cresol [43] may be represented by following equations:



Preparation of propylenediamine bis-(isatin), (PDBI) Schiff base, by the reaction of isatin with ethylenediamine can be shown as below:



PDBI reacts with tellurium (IV) chloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides in 1:1 molar ratio to form the corresponding tellurium(IV) complexes:



All the tellurium (IV) complexes are colored 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 presented in [Table 1].

Conductance Studies

The molar conductance, Λ_M at *ca.* 10^{-3} M [Table 1] for the complexes (78.68 - 96.31 Siemens $\text{cm}^2 \text{mol}^{-1}$) indicates [47, 48] that the complexes are 2:1 electrolyte type in DMSO solution. This suggests that tellurium (IV) moiety is present as dipositive cation, where as two chloride ions are as anions in the complexes. The Schiff base complexes of tellurium (IV), thus may be represented as $[\text{TeCl}_2(\text{H}_2\text{L})] \text{Cl}_2$, $[\text{RTeCl}(\text{H}_2\text{L})] \text{Cl}_2$ and $[\text{R}_2\text{Te}(\text{H}_2\text{L})] \text{Cl}_2$ respectively.

Table 1: Analytical Data, Molar Conductance and Physical Properties for Propylenediamine Bis-(Isatin) Complexes of Tellurium (IV)

Compound No.	Complex (R)	Empirical Formula (Formula Wt.)	Colour (Yield %)	M. Pt. ($^{\circ}\text{C}$)	Analyses % Found (Calculated)					Λ_M at <i>ca.</i> 10^{-3}M S $\text{cm}^2\text{mol}^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	PDBI / H_2L	$\text{C}_{19}\text{H}_{16}\text{N}_4\text{O}_2$ (332.23)	Reddish brown (90)	208-210	68.38 (68.69)	4.62 (4.82)	16.60 (16.86)	-	-	-
I	$\text{TeCl}_4(\text{H}_2\text{L})$	$\text{C}_{19}\text{H}_{16}\text{Cl}_4\text{N}_4\text{O}_2\text{Te}$ (601.64)	Orange (81)	170-172	37.67 (37.93)	2.51 (2.66)	8.99 (9.31)	20.75 (21.21)	23.04 (23.57)	96.31
II	$\text{RTeCl}_3(\text{H}_2\text{L})$ (<i>p</i> -methoxyphenyl)	$\text{C}_{26}\text{H}_{23}\text{Cl}_3\text{N}_4\text{O}_3\text{Te}$ (673.27)	Light orange (84)	176-178	45.89 (46.38)	3.11 (3.42)	7.93 (8.32)	18.30 (18.95)	15.14 (15.80)	92.34
III	$\text{RTeCl}_3(\text{H}_2\text{L})$ (<i>p</i> -hydroxyphenyl)	$\text{C}_{25}\text{H}_{21}\text{Cl}_3\text{N}_4\text{O}_3\text{Te}$ (659.26)	Reddish brown (77)	188-190	44.96 (45.55)	2.98 (3.19)	8.21 (8.50)	18.87 (19.35)	15.73 (16.13)	87.87
IV	$\text{RTeCl}_3(\text{H}_2\text{L})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{26}\text{H}_{23}\text{Cl}_3\text{N}_4\text{O}_3\text{Te}$ (673.27)	Yellowish brown (80)	204-206	46.55 (46.38)	3.09 (3.42)	8.01 (8.32)	18.36 (18.95)	15.19 (15.80)	80.61
V	$\text{R}_2\text{TeCl}_2(\text{H}_2\text{L})$ (<i>p</i> -methoxyphenyl)	$\text{C}_{33}\text{H}_{30}\text{Cl}_2\text{N}_4\text{O}_4\text{Te}$ (744.89)	Light brown (82)	135-137	54.74 (53.21)	3.88 (4.03)	6.98 (7.52)	16.68 (17.13)	9.09 (9.52)	82.74
VI	$\text{R}_2\text{TeCl}_2(\text{H}_2\text{L})$ (<i>p</i> -hydroxyphenyl)	$\text{C}_{31}\text{H}_{26}\text{Cl}_2\text{N}_4\text{O}_4\text{Te}$ (716.90)	Dark brown (78)	120-122	52.34 (51.94)	3.41 (3.63)	7.23 (7.82)	17.43 (17.80)	9.23 (9.89)	79.91
VII	$\text{R}_2\text{TeCl}_2(\text{H}_2\text{L})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{33}\text{H}_{30}\text{Cl}_2\text{N}_4\text{O}_4\text{Te}$ (744.89)	Light yellow (74)	127-129	52.79 (53.21)	3.76 (4.03)	7.06 (7.52)	16.81 (17.13)	9.11 (9.52)	78.68

Infrared Spectra

The important IR data of PDBI and its tellurium (IV) complexes are given in [Table 2]. The spectra 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 tellurium (IV) chlorides.

The three strong bands appearing at 3235, 1756 and 1648 cm^{-1} in the free PDBI ligand may be assigned [1,4,9,10] to stretching vibration modes $\nu_{(\text{N-H})}$, $\nu_{(\text{C=O})}$ and $\nu_{(\text{C=N})}$ of azomethine group, respectively.

In the IR spectra of $\text{TeCl}_4(\text{H}_2\text{L})$, $\text{RTeCl}_3(\text{H}_2\text{L})$ and $\text{R}_2\text{TeCl}_2(\text{H}_2\text{L})$ complexes, the bands of free ligand at 1756 cm^{-1} and 1648 cm^{-1} displayed shifts to lower wave numbers at 1724-1733 cm^{-1} and 1610-1618 cm^{-1} , respectively indicating the involvement of oxygen atom of C=O group of isatin residue and nitrogen atom of azomethine group in complex formation. The $\nu_{(\text{N-H})}$ band remains intact and appears at 3188-3197 cm^{-1} in the complexes. The most important conclusion drawn from infrared spectral evidence is that the propylenediamine-bis(isatin) is acting as chelating agent towards central tellurium atom as a neutral ONNO quadridentate ligand [4,6]. The $\nu_{(\text{Te-O})}$ and $\nu_{(\text{Te-N})}$ could not be ascertained due to non-availability of far infrared data.

Table 2: Important IR Data (cm⁻¹) for the Ligand (PDBI) and Complexes

Compound No.	$\nu_{(N-H)}$	$\nu_{(C=O)}$	$\nu_{(C=N)}$
PDBI / H ₂ L	3235 s	1756 mb	1648 s
I	3192 mb	1730 s	1616 s
II	3194 b	1728 s	1616 s
III	3193 s	1730 s	1617 s
IV	3194 s	1728 s	1617 s
V	3194 mb	1730 m	1618 s
VI	3188 s	1733 s	1617 s
VII	3197 s	1724 s	1610 s

s = strong, m = medium, b = broad

Proton Magnetic Resonance Spectra

The ¹H NMR spectral data of PDBI and its tellurium (IV) complexes in DMSO-d₆ are presented in [Table 3].

The spectrum of free ligand can be resolved into three distinct regions: multiplet at 2.531 - 3.341 δ ppm due to propylenic chain protons, complex multiplet at 6.816 - 7.847 δ ppm corresponding to fused aromatic nuclei of isatin residue and one singlet at 10.815 δ ppm corresponding to isatin NH residue. The integration of above ¹H NMR signals yields the proton ratio 3:4:1, which is in agreement with above assignments [4].

The proton NMR spectra of complexes i.e. TeCl₄(H₂L), RTeCl₃(H₂L) and R₂TeCl₂(H₂L) display a downfield shift of NH signal from 10.815 δ ppm to 11.051 - 11.136 δ ppm. 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 [49,50]. Also, propylenic chain bridged protons resonating at 2.531 - 3.341 δ ppm in free ligand, appear at slight downfield side (2.536 - 3.570 ppm) in the complexes, indicating the flow of electron density from the azomethine nitrogens to the tellurium centre.

Independent assignments to the aryl protons of Schiff base and RTe / R₂Te are not possible due to overlapping of signals in this region.

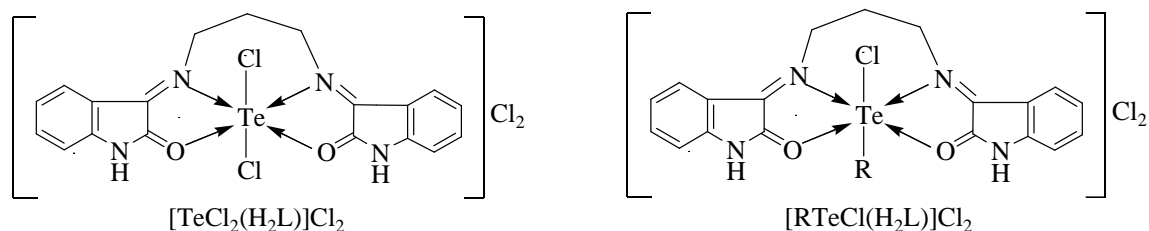
Thus, proton magnetic resonance spectral studies support the foregoing IR spectral evidence of PDBI acting as a neutral quadridentate (ONNO) ligand in these complexes.

Table 3: ¹H NMR Spectral Data of the Ligand (PDBI) and Complexes

Compound Number	Chemical Shift, δ ppm in DMSO-d ₆
PDBI / H ₂ L	2.531-3.341 (m, 6H, propylenic chain protons), 6.816-7.847 (cm, 8H, aryl protons of isatin), 10.815 (s, 2H, NH)
I	2.973-3.570 (m, 6H, propylenic chain protons), 6.924-7.549 (cm, 8H, aryl protons of isatin), 11.051 (s, 2H, NH)
II	2.939-3.425 (m, 6H, propylenic chain protons), 3.825 (s, 3H, -OCH ₃), 6.927-8.237 (cm, 12H, aryl protons of isatin and RTe), 11.101 (s, 2H, NH)
III	2.918-3.539 (m, 6H, propylenic chain protons), 6.849-8.255 (cm, 12H, aryl protons of isatin and RTe), 9.80 (bs, 1H, phenolic OH of RTe), 11.136 (s, 2H, NH)
IV	2.185 (s, 3H, CH ₃), 2.931-3.417 (m, 6H, propylenic chain protons), 6.933-8.221 (cm, 11H, aryl protons of isatin and RTe), 9.30 (bs, 1H, phenolic OH), 11.112 (s, 2H, NH)
V	2.536-3.507 (m, 6H, propylenic chain protons), 3.820 (s, 6H, OCH ₃), 6.807-8.199 (cm, 16H, aryl protons of isatin and R ₂ Te), 11.10 (s, 2H, NH)

s = singlet, m = multiplet, cm = complex multiplet, bs = broad singlet. Spectra of compound number VI and VII not well resolved due to poor solubility in DMSO-d₆

On the basis of conductance, IR and ¹H NMR spectral studies, a distorted octahedral environment around central tellurium atom may be suggested as shown in Figure 1.



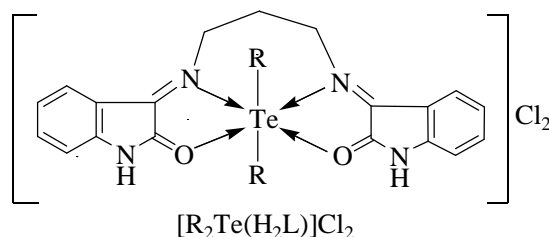


Figure 1: Proposed structures of Tellurium (IV) Propylenediamine bis-(Isatin) Complexes

Biological Studies

Propylenediamine bis-(isatin) Schiff base (PDBI) and some of its complexes were evaluated for *in vitro* antifungal and antitubercular activity.

Fungicidal activity data [Table 4] indicate that the compound III possesses better antifungal activity against all the three pathogens *i.e.* *R. solani*, *F. oxysporum* and *C. capsici*, compound V possess better antifungal activity against *F. oxysporum* and *C. capsici* and while other compounds show moderate to good activity towards these pathogens.

Antitubercular activity data were compared with the standard drug Rifampin at 0.25 $\mu\text{g/ml}$ concentrations, which showed 98% inhibition. The results are presented in [Table 5], which indicate that the compounds III were very much effective against *M. tuberculosis* at 12.5 $\mu\text{g/ml}$ concentrations and showed 91% inhibition while the other compounds showed moderate to good activity against *Mycobacterium tuberculosis*.

Table 4: Effect of Concentration of Schiff Base (PDBI) and Complexes on the Mean Radial Growth (cms) of Fungus *in vitro* (Poison Plate Technique) [44]

Compound No.	<i>Rhizoctonia solani</i>		<i>Fusarium oxysporum</i>		<i>Colletorichum capsici</i>	
	1000ppm	500ppm	1000ppm	500ppm	1000ppm	500ppm
PDBI / H ₂ L	4.55	9.00	3.67	3.67	2.31	3.56
I	5.98	8.43	1.54	3.50	3.77	4.21
III	1.42	2.69	2.16	4.17	3.25	4.58
IV	8.58	8.30	4.00	5.17	1.83	2.92
V	3.10	4.58	1.75	2.17	3.07	3.46
VI	2.10	2.25	3.68	5.28	3.58	5.98
Check	9.00	9.00	8.67	8.67	7.67	7.67
CD%	0.78	1.21	0.91	0.92	1.06	1.28

CD% = Standard antifungal drug Fluconazol

Table 5: Antitubercular Activity against *Mycobacterium tuberculosis* of Schiff Base (PDBI) and Complexes

Compound No.	Antitubercular activity	
	(H ₃₇ Rv) MIC ($\mu\text{g/ml}$)	% Inhibition
PDBI / H ₂ L	<12.5	52
I	<12.5	55
III	<12.5	91
IV	<12.5	83
V	<12.5	83
VI	<12.5	79
Rifampin	0.25	98

MIC—Minimum inhibition concentration in $\mu\text{g/ml}$

CONCLUSION

N, N'-Bis(indol -2-oxo-3-ylidene)-1,3-diaminopropane when reacted with tellurium tetrachloride, aryltellurium trichlorides and diaryltellurium dichlorides form 1:1 type complexes. Elemental analyses, conductance, infrared and proton magnetic resonance spectral studies predict the neutral ONNO quadridentate nature of ligand resulting in a distorted octahedral coordination around central tellurium atom. Some of the new tellurium (IV) complexes have been observed to possess substantial antifungal and antitubercular activity.

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