



Tellurium (IV) complexes of tridentate (ONS) Schiff base derived from isatin and 2-aminothiophenol

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ABSTRACT

Some new tellurium (IV) complexes with a tridentate Schiff base formed by condensation of isatin with 2-aminothiophenol having formulae $TeCl_3(HIATP)$, $Te(IATP)_2$, $RTeCl_2(HIATP)$ and $R_2TeCl(HIATP)$, where R = *p*-methoxy-, *p*-hydroxy- and 3-methyl-4-hydroxyphenyl and H_2IATP = Schiff base, have been synthesized and characterized by elemental analyses, conductance measurement, IR and 1H NMR spectral studies. The data predict that Schiff base acts as a monobasic tridentate (ONS) ligand in $TeCl_3(HIATP)$, $RTeCl_2(HIATP)$, $R_2TeCl(HIATP)$ and as a dibasic tridentate (ONS) ligand in $Te(IATP)_2$. Some of these complexes have been observed to possess antifungal and antitubercular activity.

Keywords: Isatin, Schiff base, Tellurium (IV), Tridentate and Antimicrobial activity.

INTRODUCTION

Isatin (1-H-indole-2, 3-dione) is an endogenous indole with a range of pharmacological actions [1-7]. A large number of Schiff bases of isatin are reported in the literature which can undergo complexation with metal ions in different modes [8-15]. Schiff base derived from isatin and 2-aminothiophenol can act as a ligand [15-17] having functional groups with nitrogen, oxygen and sulphur donor atoms.

Also, tellurium (IV) compounds such as tellurium tetrachloride [18-20], aryltellurium trichlorides [21-33] and diaryltellurium dichlorides [34-36] are known to behave as Lewis acids and form complexes with several nitrogen, oxygen and sulphur donor bases. In view of this and in continuation of earlier work on isatin Schiff bases [37-41], we herein report some new complexes of tellurium tetrachloride, aryltellurium trichlorides, $RTeCl_3$ and diaryltellurium dichlorides, R_2TeCl_2 with Isatin-2-aminothiophenol Schiff base (H_2IATP).

EXPERIMENTAL SECTION

Materials and Methods

All the chemicals were of AnalR 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 methods before use and stored over molecular sieves. *p*-Methoxyphenyltellurium(IV) trichloride [42,43], bis(*p*-methoxyphenyl)tellurium(IV) dichloride [43,44], *p*-hydroxyphenyltellurium(IV) trichloride [45], bis(*p*-hydroxyphenyl)tellurium(IV) dichloride [45], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride [46] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride [46] were prepared by the reaction of $TeCl_4$ with anisole / phenol / *o*-cresol as reported in the literature [42-46].

Carbon, hydrogen and nitrogen analyses were obtained microanalytically from SAIF, Panjab University, Chandigarh on a Perkin Elmer 2400 CHN Elemental Analyser (Thermo Scientific). Conductance measurements were performed in DMSO at $25 \pm 2^\circ C$ 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 on a F.T. Infra-Red Spectrometer Model Nicolet IS50 (Thermo Scientific). Proton NMR Spectra were recorded in DMSO- d_6 using TMS as an internal reference on BRUKER AVANCE II 400 NMR spectrometer.

Synthesis of isatin-aminothiophenol (H_2IATP) Schiff base, [(N-indol -2-oxo-3-ylidene)-2-aminothiophenol]:

The Schiff base was prepared by the method reported by Khalifa *et al* [16]. A hot ethanolic solution of isatin (0.01 mol) was added dropwise to ethanolic solution of (0.01 mol) 2-aminothiophenol under heating. The contents were refluxed on steam bath for about 3 hrs, when an orangecolored solid was precipitated. The mixture was cooled to room temperature and solid product thus obtained was filtered off, washed with ethanol, then recrystallized from DMF and dried in *vacuo* over P_4O_{10} .

Synthesis of complexes: Aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides, when treated with H_2IATP form 1:1 type of complexes whereas tellurium tetrachloride form both 1:1 and 1:2 type complexes as described below:

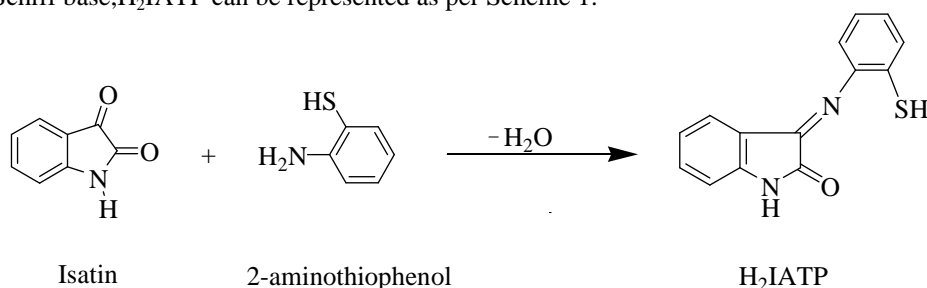
$TeCl_3$ (HIATP), R_2TeCl_2 (HIATP) and R_2TeCl (HIATP):

These types of complexes were prepared by addition of hot ethanolic solution of the Schiff base H_2IATP (10 mmol in about 50 mL) to a saturated solution of tellurium (IV) derivatives (10 mmol) in dry ethanol with continuous stirring. The reaction mixture was refluxed on steam bath for about 2 hrs. The excess solvent was distilled off to obtain the desired products, which were recrystallized from dry ethanol and dried in vacuum desiccator over P_4O_{10} . The reactions were also repeated by addition of sodium methoxide until pH 7.3, but identical products were obtained in cases of R_2TeCl_3 and R_2TeCl_2 .

$Te(IATP)_2$: Tellurium tetrachloride (5 mmol) in about 25 mL dry benzene was added to a hot saturated ethanolic solution of H_2IATP (10 mmol) with continuous stirring. Then sodium methoxide was added till pH 7.3. Contents were then stirred for about one hour when white precipitates of sodium chloride separated out, which were removed by filtration. The filtrate was refluxed for about 4 hrs and then concentrated to about one third of its original volume. This was left overnight in a refrigerator to obtain a reddish brown crystalline solid. This was filtered washed with cold dry ethanol and dried in vacuum desiccators over P_4O_{10} .

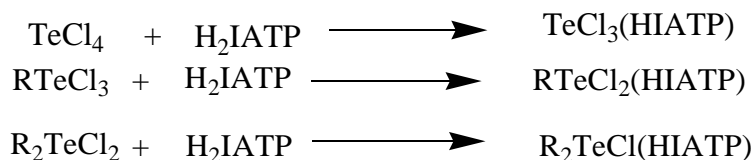
RESULTS AND DISCUSSION

Formation of Schiff base, H_2IATP can be represented as per Scheme 1:

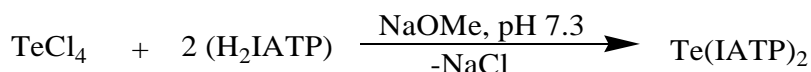


Scheme 1: Synthesis of Schiff base

This Schiff base reacts with tellurium tetrachloride, aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides in 1:1 molar ratio to yield only 1:1 type complexes:



H_2IATP reacts with tellurium tetrachloride in 2:1 molar ratio to yield the complex:



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 (11.02 – 42.88 $\text{Scm}^2 \text{mol}^{-1}$) indicate [47,48] that the complexes are non-electrolyte to weak electrolytes in DMSO solution. This may be due to donor nature of DMSO and subsequent ionization into $\text{TeCl}_2(\text{HIATP})\text{DMSO}^+ / \text{R}_2\text{TeCl}(\text{HIATP})\text{DMSO}^+ / \text{R}_2\text{Te}(\text{HIATP})\text{DMSO}^+$ and Cl^- ions in DMSO.

Table 1. Analytical data, molar conductance and physical properties for isatin-2-aminothiophenol Schiff base (H_2IATP) complexes of tellurium (IV)

Compd. No.	Complex (R)	Empirical formula (Formula wt.)	Color (Yield%)	M. Pt. ($^{\circ}\text{C}$) dec.	Analyses % Found (Calculated)					Λ_M at <i>ca.</i> 10^{-3} M $\text{Scm}^2 \text{mol}^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	H_2IATP	$\text{C}_{14}\text{H}_{10}\text{N}_2\text{OS}$ (254.23)	Orange (81)	218-220	65.89 (66.14)	4.22 (3.93)	10.49 (11.02)	-	-	-
1	$\text{TeCl}_3(\text{HIATP})$	$\text{C}_{14}\text{H}_9\text{Cl}_3\text{N}_2\text{OSTe}$ (487.18)	Light brown (78)	170-172	34.03 (34.52)	1.31 (1.85)	6.19 (5.75)	25.40 (26.19)	21.23 (21.83)	42.88
2	$\text{Te}(\text{IATP})_2$	$\text{C}_{28}\text{H}_{16}\text{N}_4\text{O}_2\text{S}_2\text{Te}$ (632.05)	Reddish brown (73)	192-194	52.74 (53.21)	2.04 (2.53)	8.22 (8.86)	19.50 (20.19)	-	11.02
3	$\text{R}_2\text{TeCl}_2(\text{HIATP})$ (<i>p</i> -methoxyphenyl)	$\text{C}_{21}\text{H}_{16}\text{Cl}_2\text{N}_2\text{O}_2\text{STe}$ (558.81)	Yellowish brown (84)	182-184	44.48 (45.14)	2.12 (2.86)	6.56 (5.01)	22.02 (22.83)	11.96 (12.69)	31.16
4	$\text{R}_2\text{TeCl}_2(\text{HIATP})$ (<i>p</i> -hydroxyphenyl)	$\text{C}_{20}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_2\text{STe}$ (544.80)	Brown (87)	166-168	43.31 (44.09)	2.01 (2.57)	4.45 (5.14)	22.77 (23.42)	12.46 (13.01)	39.14
5	$\text{R}_2\text{TeCl}_2(\text{HIATP})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{21}\text{H}_{16}\text{Cl}_2\text{N}_2\text{O}_2\text{STe}$ (558.81)	Dark brown (78)	141-143	44.59 (45.14)	2.22 (2.86)	4.32 (5.01)	22.10 (22.83)	11.98 (12.69)	29.89
6	$\text{R}_2\text{TeCl}(\text{HIATP})$ (<i>p</i> -methoxyphenyl)	$\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_3\text{STe}$ (630.43)	Pale yellow (90)	155-157	52.82 (53.35)	3.11 (3.65)	3.78 (4.44)	19.67 (20.24)	5.09 (5.62)	39.49
7	$\text{R}_2\text{TeCl}(\text{HIATP})$ (<i>p</i> -hydroxyphenyl)	$\text{C}_{26}\text{H}_{19}\text{ClN}_2\text{O}_3\text{STe}$ (602.41)	Light brown (86)	129-131	51.33 (51.84)	2.68 (3.15)	4.01 (4.65)	20.55 (21.18)	6.43 (5.89)	36.19
8	$\text{R}_2\text{TeCl}(\text{HIATP})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_3\text{STe}$ (630.43)	Dark brown (82)	138-140	52.73 (53.35)	3.17 (3.65)	3.86 (4.44)	19.54 (20.24)	5.01 (5.62)	41.76

Values of Λ_M reported [47,48] for 1:1 electrolyte in DMSO = 50 – 70 $\text{Scm}^2 \text{mol}^{-1}$

Infrared spectra

The important infrared spectral data of H_2IATP and its tellurium (IV) complexes are compiled in Table 2. The spectra of tellurium (IV) complexes are quite complex and thus, an attempt has been made to identify the donor sites of the Schiff base by comparing the spectra of complexes with the parent ligand and tellurium (IV) chlorides.

The three strong bands appearing at 3186, 1726 and 1618 cm^{-1} in the ligand spectra may be assigned [16] to stretching vibration modes $\nu_{\text{N-H}}$, $\nu_{\text{C=O}}$ and $\nu_{\text{C=N}}$ respectively. The $\nu_{\text{S-H}}$ in free ligand appears at 2605 cm^{-1} as a weak band.

In the IR spectra of 1:1 complexes, i.e. $\text{TeCl}_3(\text{HIATP})$, $\text{R}_2\text{TeCl}_2(\text{HIATP})$ and $\text{R}_2\text{TeCl}(\text{HIATP})$, the bands of free ligand at 1726 cm^{-1} and 1618 cm^{-1} displayed shifts to lower wave numbers at 1700 - 1712 cm^{-1} and 1575 - 1597 cm^{-1} , respectively indicating the involvement of oxygen atom of C=O group of isatin residue and nitrogen atom of azomethine group in the complex formation. The $\nu_{\text{N-H}}$ band remains intact and appears at 3169 - 3197 cm^{-1} in the complexes. The $\nu_{\text{S-H}}$, which appears at 2605 cm^{-1} in the parent ligand, disappears in all these complexes indicating thereby the deprotonation of SH group of Schiff base and subsequent linkage of sulphur to the tellurium atom.

The most important conclusion drawn from infrared spectral evidence is that the isatin-2-aminothiophenol is acting as chelating agent towards central tellurium atom as a uninegative (ONS) tridentate ligand [16] through central azomethine nitrogen and sulphur of SH (after deprotonation) and oxygen of carbonyl group. The $\nu_{\text{Te-O}}$, $\nu_{\text{Te-N}}$ and $\nu_{\text{Te-S}}$ could not be ascertained due to non-availability of far infrared data.

In 1:2 complex, $\text{Te}(\text{IATP})_2$, all the bands assigned to $\nu_{\text{N-H}}$, $\nu_{\text{C=O}}$ and $\nu_{\text{C=N}}$ in the free ligand, disappear and new strong bands are observed at 1493 cm^{-1} and 1176 cm^{-1} . These may be assigned to new azomethine $\nu_{\text{C=N}}$ due to enolization of NH hydrogen of isatin and $\nu_{\text{C-O}}$ vibration after coordination at tellurium through oxygen of C-O group. Thus, H_2IATP Schiff base ligand is coordinated to central tellurium atom as binegative (ONS) tridentate ligand.

Table 2. Important IR data (cm⁻¹) of Schiff base (H₂IATP) and complexes

Compound No.	$\nu_{(N-H)}$	$\nu_{(C=O)}$	$\nu_{(C=N)}$	$\nu_{(S-H)}$
H ₂ IATP	3186 mb	1726 s	1618 s	2605 m
1	3169 s	1700 s	1579 s	-
2	-	-	1584 s, 1493 s ^a	-
3	3197 mb	1712 s	1581 s	-
4	3192 mb	1710 sh	1575 sh	-
5	3183 mb	1705 s	1597 s	-
6	3188 mb	1712 s	1582 sh	-
7	3190 mb	1706 sh	1591 s	-
8	3177 mb	1710 sh	1578 s	-

s = strong, *m* = medium, *b* = broad, *sh* = shoulder, *a* = $\nu_{C=N}$

Proton magnetic resonancespectra

The ¹H NMR spectra of free ligand and its tellurium (IV) complexes were measured in DMSO-d₆ and the data are presented in Table 3.

The spectrum of free ligand can be resolved into four distinct regions, complex multiplets at 6.550- 7.047 δ ppm and 7.160 - 7.568 δ ppm corresponding to aryl proton of aminothiophenol and isatin skeleton, one singlet at 4.328 δ ppm corresponding to SH of Schiff base and a singlet at 11.019 δ ppm due to isatin NH residue.

The proton NMR spectra of 1:1 complexes i.e. TeCl₃(HIATP), RTeCl₂(HIATP) and R₂TeCl(HIATP) display a downfield shift from 11.019 to 11.035 - 11.060 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 [16,49,50]. Also, SH proton resonating at 4.328 δ ppm in free ligand, disappears in all the complexes including 1:2 i.e. Te (IATP)₂ complexes, thereby indicating its deprotonation and subsequently linkage to tellurium as predicted by IR spectra as well. In Te (IATP)₂, NH proton also disappears, further supporting the enolization of this proton as exhibited in IR spectra.

Table 3. ¹H NMR spectral data of Schiff base (H₂IATP) and complexes

Compound No.	Chemical Shift, δ ppm in DMSO-d ₆
H ₂ IATP	6.550-7.047 (cm, 4H, aryl protons of aminothiophenol), 7.160-7.568 (cm, 4H, aryl protons of isatin moiety), 4.328 (s, 1H, -SH of aminothiophenol), 11.019 (s, 1H, NH)
1	6.909-7.583 (cm, 8H, aryl protons of Schiff base), 11.049 (s, 1H, NH)
2	6.913-7.565 (cm, 16H, aryl protons of Schiff base)
3	3.21 (s, 3H, -OCH ₃), 6.912-7.587 (cm, 12H, aryl protons of Schiff base & RTe), 11.060 (s, 1H, NH)
4	6.825-7.782 (cm, 12H, aryl protons of Schiff base & RTe), 8.258 (s, 1H, phenolic OH of RTe), 11.048 (s, 1H, NH)
5	2.526 (s, 3H, -CH ₃), 6.904-7.902 (cm, 11H, aryl protons of Schiff base & RTe), 8.381 (s, 1H, phenolic OH of RTe), 11.051 (s, 1H, NH)
6	3.443 (s, 6H, -OCH ₃), 6.853-7.586 (cm, 16H, aryl protons of Schiff base and R ₂ Te), 11.052 (s, 1H, NH)
7	6.688-7.680 (cm, 16H, aryl protons of Schiff base and R ₂ Te), 8.163 (bs, 2H, phenolic OH of R ₂ Te), 11.035 (s, 1H, NH)
8	2.533 (s, 6H, -CH ₃), 6.904-7.574 (cm, 14H, aryl protons of Schiff base and R ₂ Te), 8.163 (bs, 2H, phenolic OH of R ₂ Te), 11.039 (s, 1H, NH)

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

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 H₂IATP acting as a monobasic uninegative tridentate (ONS) ligand in 1:1 complexes and as a dibasic binegative tridentate (ONS) ligand in 1:2 type complex.

On the basis of spectral studies, a distorted octahedral environment around central tellurium atom may be suggested as shown in Fig. 1.

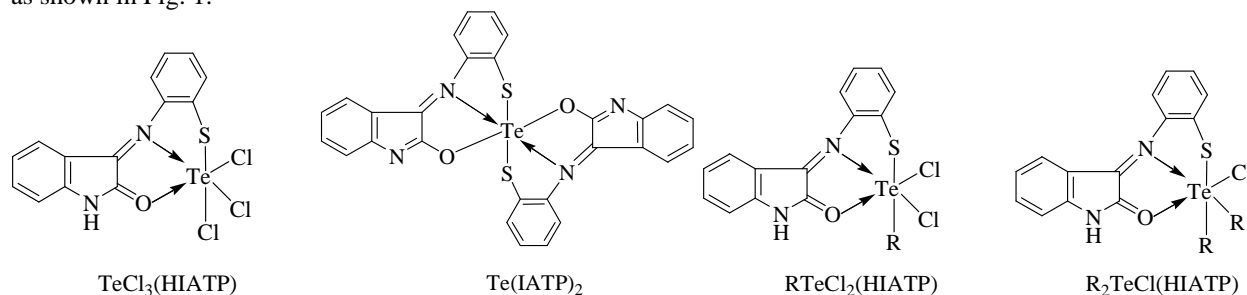


Fig. 1. Proposed structures of tellurium (IV) isatin-aminothiophenol Schiff base complexes

Biological studies

For antifungal activity measurements, the compounds were prepared in 1000 and 500 ppm concentrations in acetone using Poison Plate Technique method [51]. 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 [52,53]. 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 $\mu\text{g}/\text{mL}$.

Isatin-aminothiophenol Schiff base (H₂IATP) and some of its complexes were evaluated for antifungal and antitubercular activity *in vitro*. Fungicidal activity data, Table 4, indicate that the compounds 3 and 7 possess better antifungal activity against all the three pathogens and all compounds except Schiff base show better activity against *C. capsici* and *F. oxysporum* fungi while other compounds show moderate to good activity towards these pathogens. In general, the antifungal activity of complexes towards fungi decreases in the order

C. capsici \geq *F. oxysporum* $>$ *R. solani*.

Table 4. Effect of concentration of Schiff base (H₂IATP) and complexes on the mean radial growth (cms) of fungus *in vitro* (Poison Plate Technique) [51]

Compound No.	<i>Rhizoctonia solani</i>		<i>Fusarium oxysporum</i>		<i>Colletorichum capsici</i>	
	1000ppm	500ppm	1000ppm	500ppm	1000ppm	500ppm
H ₂ IATP	6.48	5.21	3.22	5.36	5.33	7.11
1	6.53	7.23	1.36	2.45	4.22	6.32
3	1.12	2.39	1.05	4.02	1.10	2.36
4	7.13	7.52	1.36	2.82	2.36	4.09
5	8.02	8.45	3.32	4.13	2.66	6.69
7	1.36	1.42	1.69	3.35	1.39	2.98
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

Antitubercular activity data were compared with the standard drug Rifampin at 0.25 $\mu\text{g}/\text{mL}$ concentrations, which showed 98% inhibition. The results are presented in Table 5, which indicate that the compounds 3, 5 and 7 were very much effective against *M. tuberculosis* at 12.5 $\mu\text{g}/\text{mL}$ concentrations and showed 91–94% inhibition while the other compounds showed moderate to good activity against *Mycobacterium tuberculosis*.

Table 5. Antitubercular Activity against *Mycobacterium tuberculosis* of Schiff base (H₂IATP) and complexes

Compound No.	Antitubercular activity	
	(H ₃₇ Rv) MIC ($\mu\text{g}/\text{mL}$)	% Inhibition
H ₂ IATP	<12.5	56
1	<12.5	53
3	<12.5	94
4	<12.5	87
5	<12.5	91
7	<12.5	92
Rifampin	0.25	98

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

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