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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES ON SOME COMPLEXES OF TELLURIUM (IV) WITH SCHIFF BASE DERIVED FROM ISATIN AND 2-AMINOPHENOL

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ABSTRACT

Eight new tellurium(IV) chelates with a tridentate Schiff base formed by condensation of isatin with 2-aminophenol having formulae $TeCl_3$(HIAP), $Te(IAP)_2$, $RTeCl_2$(HIAP) and R_2TeCl(HIAP); where R = *p*-methoxy-, *p*-hydroxy- and 3-methyl-4-hydroxyphenyl and H_2IAP = Schiff base, have been synthesized and characterized *via* elemental analyses, conductance measurement, infrared and proton magnetic resonance spectral studies. The data predict that Schiff base acts as a monobasic tridentate (ONO) ligand in $TeCl_3$(HIAP), $RTeCl_2$(HIAP), R_2TeCl(HIAP) and as a dibasic tridentate (ONO) ligand in $Te(IAP)_2$. Some of these complexes have also been observed to possess antifungal and antitubercular activity.

Key words: 2-Aminophenol, Isatin, Schiff base, Tellurium (IV), Tridentate, Antimicrobial activity.

INTRODUCTION

Isatin is an endogenous indole with a range of pharmacological actions[1-7]. A number of Schiff's bases of isatin are reported in the literature, which undergoes complexation with metal ions in different modes[8-15]. Schiff base derived from isatin and 2-aminophenol can act as a ligand[8,9,15] having functional groups with nitrogen and oxygen donor atoms.

Also, tellurium (IV) compounds such as tellurium tetrachloride[16-18], aryltellurium trichlorides[19-31] and diaryltellurium dichlorides[32-34] 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[35-38], we herein report some new

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complexes of tellurium (IV) chloride, aryltellurium trichlorides, $RTeCl_3$ and diaryltellurium dichlorides, R_2TeCl_2 with isatin-2-aminophenol Schiff base (H_2IAP).

EXPERIMENTAL

Materials and methods

All the chemicals were of Anal R 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[39,40], bis(*p*-methoxyphenyl)tellurium(IV) dichloride[40,41], *p*-hydroxyphenyl tellurium (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 $TeCl_4$ with anisole /phenol /*o*-cresol as reported in the literature[39-43].

Preparation of isatin-aminophenol (H_2IAP) Schiff base, [(N-indol-2-oxo-3-ylidene)-2-aminophenol]

The Schiff base was prepared by the method reported by Kriza et al.[9] A hot methanolic solution of isatin (0.02 mol; 2.94 g) was added to methanolic solution of (0.02 mol; 2.18 g) 2-aminophenol under heating. The contents were refluxed for 4 hrs during, which time; a brown product was precipitated. The mixture was cooled to room temperature and solid product thus obtained was filtered off, washed with methanol and dried in vacuo over P_4O_{10}.

Synthesis of complexes

Aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides, when treated with H_2IAP form 1:1 type of complexes whereas tellurium tetrachloride form both 1:1 and 1:2 type complexes as described below:

$TeCl_3$(HIAP), $RTeCl_2$(HIAP) and R_2TeCl(HIAP)

These types of complexes were prepared by addition of hot methanolic solution of the Schiff base H_2IAP (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 about 4 hrs. The excess solvent was distilled off to obtain the desired products, which were recrystallized from dry methanol 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 $RTeCl_3$ and R_2TeCl_2.

$Te(IAP)_2$

Tellurium tetrachloride (5 mmol) in about 25 mL dry benzene was added to a saturate hot methanolic solution of H_2IAP (10 mmol) with continuous stirring. Then sodium methoxide was added till pH 7.3. Contents were then stirred for about one hour when white precipitated of sodium chloride separated out, which were removed by filtration. The filtrate was refluxed for about 6 hrs and then concentrated to about one third of original volume. This was left overnight in a refrigerator to obtain a grey colored crystalline solid. This was filtered washed with cold dry methanol and dried in a vacuum desiccators over P_4O_{10}.

Physical studies

Carbon, hydrogen and nitrogen analyses were obtained microanalytically from Sophisticated Analytical Instrumentation Facility, Panjab University, Chandigarh on a Perkin Elmer 2400 CHN Elemental Analyser (Thermo Scientific). Conductance measurements were performed in DMSO at 25±2˚C with a dip type conductivity cell (cell constant = 1.017) on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra (4000-400 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.

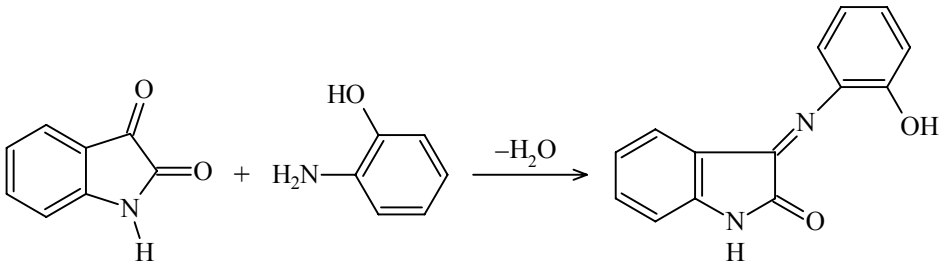
Antimicrobial studies

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. Then the measurements were carried out by methods as described earlier[37,38].

Antitubercular activity was evaluated in DMSO against *M. tuberculosis* $H_{37}Rv$ 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 μg/ml.

RESULTS AND DISCUSSION

Formation of isatin-aminophenol, (H_2IAP) Schiff base, by the reaction of isatin with 2-aminophenol can be represented by following equation:



Isatin 2-Aminophenol H_2IAP

H_2IAP reacts with tellurium tetrachloride in 1:1 and 2:1 molar ratios to yield the complexes:

$$TeCl_4 + H_2IAP \xrightarrow{-HCl} TeCl_3(HIAP)$$

$$TeCl_4 + 2\,(H_2IAP) \xrightarrow[-NaCl]{NaOMe,\ pH\ 7.3} Te\,(IAP)_2$$

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

$$RTeCl_3 + H_2IAP \longrightarrow RTeCl_2\,(HIAP)$$

$$R_2TeCl_2 + H_2IAP \longrightarrow R_2TeCl\,(HIAP)$$

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 (9.11-34.18 S cm^2 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 $TeCl_2(HIAP)DMSO^+$/$RTeCl(HIAP)DMSO^+$/$R_2Te(HIAP)DMSO^+$ and Cl^- ions in DMSO.

Infrared spectra

The important infrared spectral data of H_2IAP 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 ligand by comparing the spectra of complexes with the parent ligand and tellurium (IV) chlorides.

Table 1: Analytical data, molar conductance and physical properties for isatin- 2-aminophenol Schiff base (H_2IAP) complexes of tellurium (IV)

Compd. 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	H_2IAP	$C_{14}H_{10}N_2O_2$ (238.17)	Brown (87)	192-194	70.02 (70.60)	4.59 (4.20)	11.24 (11.76)	-	-	-
1	$TeCl_3$(HIAP)	$C_{14}H_9Cl_3N_2O_2Te$ (471.12)	Dark brown (81)	187-189	36.05 (35.69)	1.61 (1.91)	6.47 (5.95)	26.34 (27.08)	22.01 (22.58)	34.18
2	Te $(IAP)_2$	$C_{28}H_{16}N_4O_4Te$ (599.93)	Dark grey (84)	172-174	55.47 (56.06)	2.21 (2.67)	8.69 (9.34)	20.61 (21.27)	-	9.11
3	$RTeCl_2$(HIAP) (*p*-methoxyphenyl)	$C_{21}H_{16}Cl_2N_2O_3Te$ (542.75)	Light brown (72)	148-150	45.94 (46.47)	3.41 (2.95)	4.55 (5.16)	22.91 (23.51)	12.55 (13.06)	26.88
4	$RTeCl_2$(HIAP) (*p*-hydroxyphenyl)	$C_{20}H_{14}Cl_2N_2O_3Te$ (528.74)	dark brown (81)	122-124	44.88 (45.43)	2.21 (2.65)	4.79 (5.30)	23.63 (24.13)	12.69 (13.41)	27.18
5	$RTeCl_2$(HIAP) (3-methyl-4-hydroxyphenyl)	$C_{21}H_{16}Cl_2N_2O_3Te$ (542.75)	Grayish brown (86)	128-130	45.77 (46.47)	3.46 (2.95)	4.59 (5.16)	23.01 (23.51)	12.58 (13.06)	32.54
6	R_2TeCl(HIAP) (*p*-methoxyphenyl)	$C_{28}H_{23}ClN_2O_4Te$ (614.37)	Light brown (77)	116-118	54.13 (54.74)	3.11 (3.74)	4.01 (4.56)	20.11 (20.77)	5.23 (5.77)	21.19
7	R_2TeCl(HIAP) (*p*-hydroxyphenyl)	$C_{26}H_{19}ClN_2O_4Te$ (586.35)	Blackish (88)	162-164	52.75 (53.26)	2.87 (3.24)	4.05 (4.78)	21.08 (21.76)	6.65 (6.05)	32.67
8	R_2TeCl(HIAP) (3-methyl-4-hydroxyphenyl)	$C_{28}H_{23}ClN_2O_4Te$ (614.37)	Brown (76)	136-138	54.19 (54.74)	3.16 (3.74)	3.97 (4.56)	21.96 (20.77)	5.34 (5.77)	31.28

Values of Λ_M reported[47,48] for 1:1 electrolytes in DMSO = 50 – 70 S cm^2 mol^{-1}

Table 2: Important IR data (cm^{-1}) of the ligand (H_2IAP) and complexes

Compd. No.	$\upsilon_{(N-H)}$	$\upsilon_{(C=O)}$	$\upsilon_{(C=N)}$	$\upsilon_{(O-H)}$
H_2IAP	3136 mb	1732 s	1673 s	3251 mb
1	3124 s	1710 s	1635 s	-
2	-	-	1615 s, 1559 s[a]	-
3	3113 mb	1710 s	1615 s	-
4	3110 mb	1712 s	1616 s	-
5	3104 mb	1715 s	1615 s	-
6	3110 mb	1716 s	1616 s	-
7	3117 s	1719 s	1614 s	-
8	3112 mb	1713 sh	1618 s	-

The three strong bands appearing at 3136, 1732 and 1673 cm^{-1} in the ligand spectra may be assigned[8,9] to stretching vibration modes υ_{N-H}, $\upsilon_{C=O}$ and $\upsilon_{C=N}$, respectively.

In the IR spectra of 1:1 complexes, i.e. $TeCl_3$(HIAP), $RTeCl_2$(HIAP) and R_2TeCl (HIAP), the bands of free ligand at 1732 cm^{-1} and 1673 cm^{-1} displayed shifts to lower wave numbers 1710-1719 cm^{-1} and 1614-1635 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 υ_{N-H} band remains intact and appears at 3104-3124 cm^{-1} in the complexes. The υ_{O-H}, which appears at 3251 cm^{-1} in the parent ligand, disappears in all these complexes indicating thereby the deprotonation of phenolic OH group of Schiff base.

The υ_{O-H} in complexes of hydroxyaryl tellurium (IV) derivatives appears relatively at higher wave numbers (3350-3500 cm^{-1}) as distinct broad bands. The most important conclusion drawn from infrared spectral evidence is that the isatin-aminophenol is acting as chelating agent towards central tellurium atom as a uninegative *ONO* tridentate ligand[9,49] through central azomethine nitrogen and two oxygens of hydroxyl (after deprotonation) and carbonyl group. The υ_{Te-O} and υ_{Te-N} could not be ascertained due to non-availability of Far IR data.

In 1:2 complex, $Te(IAP)_2$, the bands assigned to υ_{N-H} and $\upsilon_{C=O}$ in the free ligand, disappear and new strong bands are observed at 1559 cm^{-1} and 1203 cm^{-1}. These may be

assigned to new azomethine $\upsilon_{C=N}^{*}$ due to enolization of NH hydrogen of isatin and υ_{C-O} vibration after coordination at tellurium through oxygen of C-O group. Azomethine, $\upsilon_{C=N}$ shifts to 1615 cm^{-1} due to coordination through this nitrogen atom. Thus, H_2IAP Schiff base ligand is coordinated to central tellurium atom as binegative (*ONO*) tridentate ligand as in cases of Sn(IV) and Zr(IV) complexes[9].

Proton magnetic resonance spectra

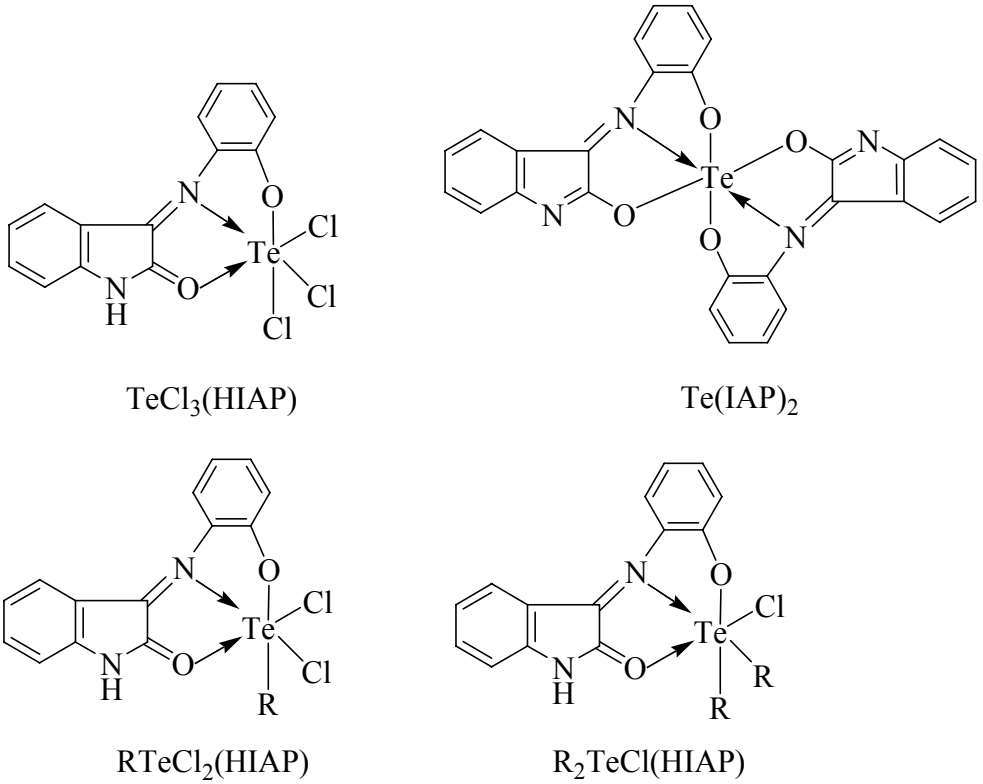
The ^1H NMR spectra of free ligand and its tellurium (IV) complexes were measured in DMSO-d_6 and the data are presented in Table 3.

Table 3: ^1H NMR spectral data of Schiff base (H_2IAP) and complexes

Compd. No.	Chemical Shift, δ ppm in DMSO-d_6
H_2IAP	6.621-6.965 (cm, 4H, aryl protons of aminophenol), 7.286-7.400 (cm, 4H, aryl protons of isatin moiety), 9.261 (s, 1H, phenolic OH of aminophenol), 10.874 (s, 1H, NH)
1	6.427-7.574 (cm, 8H, aryl protons of Schiff base), 10.921 (s, 1H, NH)
2	6.458-7.460 (cm, 16H, aryl protons of Schiff base)
3	3.39 (s, 3H, -OCH_3), 6.379-7.476 (cm,12H, aryl protons of Schiff base & RTe), 11.019 (s,1H, NH)
4	6.386-7.590 (cm, 12H, aryl protons of Schiff base & RTe), 8.217 (s, 1H, phenolic OH of RTe), 11.001 (s, 1H, NH)
5	2.169 (s, 3H, -CH_3), 6.670-7.676 (cm, 11H, aryl protons of Schiff base & RTe), 8.225 (s, 1H, phenolic OH of RTe), 11.087 (s, 1H, NH)
6	3.416 (s, 6H, -OCH_3), 6.424-7.583 (cm, 16H, aryl protons of Schiff base and R_2Te), 11.073 (s, 1H, NH)
7	6.684-7.798 (cm, 16H, aryl protons of Schiff base and R_2Te), 8.292 (bs, 2H, phenolic OH of R_2Te), 11.043 (s, 1H, NH)
8	2.168 (s, 6H, -CH_3), 6.853-7.675 (cm, 14H, aryl protons of Schiff base and R_2Te), 8.230 (bs, 2H, phenolic OH of R_2Te), 11.071 (s, 1H, NH)

s = Singlet, cm = Complex multiplet, bs = Broad singlet

The spectrum of free ligand can be resolved into four distinct regions: complex multiplets at 6.621-6.965 δ ppm and 7.286-7.400 δ ppm corresponding to aryl proton of aminophenol and isatin skeleton, one singlet at 9.261 δ ppm corresponding to phenolic OH of Schiff base and a singlet at 10.874 δ ppm due to isatin NH residue.



$TeCl_3(HIAP)$ $Te(IAP)_2$

$RTeCl_2(HIAP)$ $R_2TeCl(HIAP)$

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

The proton NMR spectra of 1:1 complexes i.e. $TeCl_3(HIAP)$, $RTeCl_2(HIAP)$ and $R_2TeCl(HIAP)$ display a downfield shift from 10.874 to 10.921-11.087 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[49-51]. Also, phenolic OH proton resonating at 9.261 δ ppm in free ligand, disappears in all the complexes including 1:2 i.e. $Te(IAP)_2$ complexes, thereby indicating its deprotonation and subsequently linkage to tellurium as predicted by IR spectra as well. In $Te(IAP)_2$, NH proton also disappears, further supporting the enolization of this proton as exhibited in IR spectra.

Independent assignments to the aryl protons of Schiff base and RTe/R_2Te are not possible due to overlapping of signals in this region. Thus, proton magnetic resonance spectral studies support the foregoing IR spectral evidence of H_2IAP acting as a monobasic uninegative tridentate (*ONO*) ligand in 1:1 complexes and dibasic binegative tridentate (*ONO*) ligand in 1:2 type complexes. On the basis of spectral studies, a distorted octahedral environment around central tellurium atom may be suggested as shown in Fig. 1.

Biological studies

Isatin-aminophenol Schiff base (H_2IAP) and some of its complexes were evaluated for antifungal and antitubercular activity *in vitro.* Fungicidal activity data (Table 4) indicate that the compound 7 possesses better antifungal activity against all the three pathogens and all compounds except Schiff base show better activity against *C. capsici* fungus 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 > *F. oxysporum* > *R. solani*

Table 4: Effect of concentration of Schiff base (H_2IAP) and complexes on the mean radial growth (cms) of fungus *in vitro* (Poison Plate Technique)[44]

Compd. No.	*Rhizoctonia solani*		*Fusarium oxysporum*		*Colletorichum capsici*	
	1000 ppm	**500 ppm**	**1000 ppm**	**500 ppm**	**1000 ppm**	**500 ppm**
H_2IAP	7.92	8.68	3.25	4.33	3.25	4.35
1	6.52	6.54	3.17	3.58	**1.55**	**2.95**
3	8.30	9.00	3.00	4.00	**3.00**	4.81
4	9.00	9.00	3.75	4.25	**2.67**	**3.79**
5	**1.75**	**2.75**	4.25	4.33	**1.35**	**2.08**
7	**1.33**	8.02	**2.50**	4.25	**2.83**	4.33
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 µg/mL concentrations, which showed 98% inhibition. The results are presented in Table 5, which indicate that the compounds 4 and 7 were very much effective against

M. tuberculosis at 12.5 μg/mL concentrations and showed 92-94% inhibition while the other compounds showed moderate to good activity against *Mycobacterium tuberculosis*.

Table 5: Antitubercular activity against *mycobacterium tuberculosis* of Schiff base (H_2IAP) and complexes

Compound No.	Antitubercular activity	
	(H_{37}Rv) MIC (μg/mL)	% Inhibition
H_2IAP	< 12.5	53
1	< 12.5	59
3	< 12.5	72
4	< 12.5	**92**
5	< 12.5	81
7	< 12.5	**94**
Rifampin	0.25	98

MIC-Minimum inhibition concentration in μg/mL

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