

Complexes of Tellurium(IV) with Isatin-Aniline Schiff Base

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Abstract: A series of new tellurium(IV) complexes with Schiff base of isatin (1-*H*-indol-2,3-dione) with aniline 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 =4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl and HL =HIAN. These complexes have been characterized by elemental analyses, conductance measurement, FT-IR and ^1H NMR spectral studies. The 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 geometry.

Keywords: Isatin-Aniline Schiff base, Tellurium tetrachloride, Aryltellurium(IV), Diaryltellurium(IV), Bidentate

Introduction

Isatin (1-*H*-indol-2,3-dione) is a resourceful endogenous heterocyclic molecule identified in human being and several of its derivatives are reported to exhibit a range of promising pharmacodynamic profile like anticonvulsant^{1,2}, anti-HIV³, cytotoxic⁴, tuberculostatic⁵ and antimicrobial⁶. The biological and pharmacological properties of isatin have been reviewed by da Silva *et al*⁷. A large number of Schiff's bases of isatin have been reported in the literature which undergoes complexation with various metal ions in different modes⁸⁻¹⁶. Schiff bases derived from isatin with aniline can act as ligand⁹⁻¹³ having functional groups with nitrogen and oxygen donor atoms.

Also, aryltellurium(IV) trichlorides are known¹⁷⁻³⁰ 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) trichlorides³¹⁻³³. Tellurium(IV) chloride is also known to form adducts with amides³⁴⁻³⁵ and thiourea³⁶, thus reflecting its acceptor behaviour. In view of this, we have investigated the reactions of tellurium(IV) chloride, some aryltellurium trichlorides, RTeCl_3 and diaryltellurium dichlorides, R_2TeCl_2 with Isatin-aniline Schiff base (HIAN), to synthesize some new complexes of tellurium(IV).

Experimental

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^{37,38} before use. The purity of compounds was checked by TLC 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. 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 ($4000\text{--}400\text{ cm}^{-1}$) were recorded in KBr pellets at SAIF, Panjab University Chandigarh on a F.T. Infra-Red spectrophotometer Model RZX (Perkin Elmer). ^1H NMR spectra were recorded in DMSO- d_6 using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer.

Preparation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides

4-Methoxyphenyltellurium(IV) trichloride^{39,40}, bis(4-methoxyphenyl)tellurium(IV) dichloride^{40,41}, 4-hydroxyphenyltellurium(IV) trichloride⁴², bis(4-hydroxyphenyl) tellurium(IV) dichloride⁴², 3-methyl-4-hydroxyphenyltellurium(IV) trichloride⁴³ and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride⁴³ 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^{39,43}.

Preparation of Isatin - Aniline Schiff Base (HIAN)

The Schiff base has been prepared by method reported by Kriza and Parnau¹⁵. Equimolar quantity of isatin and aniline were dissolved in dry ethanol and refluxed for 3 hours. After completion of reaction, the reaction mixture was kept overnight to get the solid product. The product was filtered, dried and recrystallized from ethanol. Yield = 86%, M. Pt. = $214\text{--}216$ °C. Analyses (calculated %) $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}$: C (75.69), H (4.50) and N (12.61); found: C 75.35, H 4.65 and N 12.42.

Preparation of complexes

Tellurium tetrachloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides, when reacted with Isatin-Aniline Schiff base (HIAN) form two types of complexes under different experimental conditions as described below:

TeCl_4 (HL), RTeCl_3 (HL) and R_2TeCl_2 (HL)

These types of complexes were prepared by addition of tellurium(IV) derivatives (5 mmol) in about 25 mL anhydrous methanol to a hot methanolic solution of the HIAN (5 mmol in about 25 mL) 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.

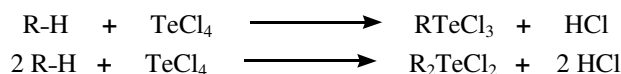
TeCl_2 (L)₂, RTeCl (L)₂, R_2Te (L)₂

These complexes were prepared by addition of tellurium(IV) compounds *i.e.* TeCl_4 / RTeCl_3 / R_2TeCl_2 (5 mmol) in 25 mL anhydrous methanol to a hot methanolic solution of the HIAN (10 mmol / 50 mL) with continuous stirring. Sodium methoxide was then added until pH reached to about 7.4. Stirred for about 1 hour and removed white turbidity by filtration.

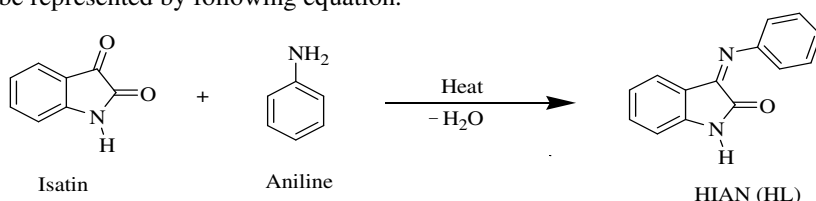
The reaction mixture was then refluxed on steam bath for about 6 hours. The excess solvent was distilled off to about one third of original volume and kept at room temperature overnight. The coloured complexes crystallized out, which were filtered, washed with dry methanol and dried in a vacuum desiccator over P_4O_{10} .

Results and Discussion

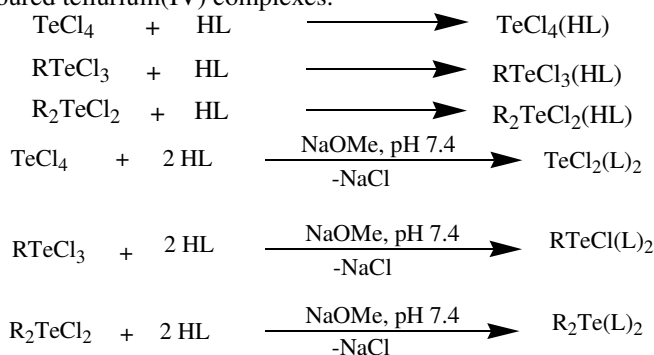
Formation of aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides by the reaction of tellurium tetrachloride with anisole³⁹⁻⁴¹, phenol⁴² and *o*-cresol⁴³ (R-H) involves the electrophilic substitution of the aromatic ring by a trichlorotellurium group at a position *para* to the methoxy / hydroxyl group.



Preparation of Isatin - Aniline Schiff Base (HIAN), by the reaction of isatin with aniline can be represented by following equation.



Isatin- Aniline Schiff Base (HIAN) reacts with tellurium(IV) chloride, aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides in 1:1 (neutral) and 2:1 (basic) molar ratios 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 and physical properties of ligand and the complexes are recorded in Table 1.

Conductance studies

Molar conductance (Λ_M) data for the complexes in DMSO are compiled in Table 1. The Λ_M values at *ca.* 10^{-3} M predict the non-electrolyte to 1:1 electrolyte^{44,45} 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 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.

Table 1. Analytical Data, Molar Conductance and Physical Properties for Isatin-Aniline Schiff Base (HIAN) Complexes of Tellurium(IV)

Compd No.	Complex (R)	Empirical formula (Formula Wt.)	Colour, (Yield, %)	M.P. °C	Analyses % Found (Calculated)					Λ_M at <i>ca.</i> $10^{-3}M$ $ohm^{-1} cm^2 mol^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	HIAN or HL	$C_{14}H_{10}N_2O$ (222.17)	Yellow orange (86)	214-216	75.35 (75.69)	4.65 (4.50)	12.42 (12.61)	-	-	-
I	$TeCl_4$ (HL)	$C_{14}H_{10}Cl_4N_2OTe$ (491.58)	Red (82)	224-226	33.94 (34.21)	2.25 (2.03)	5.42 (5.70)	25.84 (25.96)	28.42 (28.85)	46.38
II	$TeCl_2$ (L) ₂	$C_{28}H_{18}Cl_2N_4O_2Te$ (640.84)	Light Yellow (79)	204-206	52.20 (52.48)	3.02 (2.81)	8.52 (8.74)	19.74 (19.91)	11.23 (11.06)	20.15
III	$RTeCl_3$ (HL) (4-methoxyphenyl)	$C_{21}H_{17}Cl_3N_2O_2Te$ (563.20)	Grey Brown (80)	194-196	44.45 (44.79)	3.24 (3.02)	4.81 (4.97)	22.40 (22.66)	18.53 (18.88)	47.70
IV	$RTeCl(L)_2$ (4-methoxyphenyl)	$C_{35}H_{25}ClN_4O_3Te$ (712.46)	Yellow (82)	201-203	58.85 (59.00)	3.65 (3.51)	7.76 (7.86)	17.79 (17.91)	4.72 (4.98)	71.90
V	$RTeCl_3$ (HL) (4-hydroxyphenyl)	$C_{20}H_{15}Cl_3N_2O_2Te$ (549.19)	Dark brown(90)	177-179	43.52 (43.74)	2.79 (2.73)	4.94 (5.10)	23.07 (23.23)	19.14 (19.37)	6.44
VI	$RTeCl(L)_2$ (4-hydroxyphenyl)	$C_{34}H_{23}ClN_4O_3Te$ (698.45)	Light orange(88)	181-183	58.22 (58.47)	3.42 (3.29)	7.91 (8.02)	18.40 (18.27)	4.92 (5.08)	68.89
VII	$RTeCl_3$ (HL) (3-methyl-4-hydroxyphenyl)	$C_{21}H_{17}Cl_3N_2O_2Te$ (563.20)	Orange (79)	132-134	44.27 (44.77)	3.44 (3.04)	4.47 (4.97)	22.15 (22.65)	18.46 (18.88)	47.80
VIII	$RTeCl(L)_2$ (3-methyl-4-hydroxyphenyl)	$C_{35}H_{25}ClN_4O_3Te$ (712.46)	Light brown (81)	207-209	59.39 (58.99)	2.94 (3.54)	7.26 (7.86)	17.30 (17.90)	4.57 (4.97)	56.04
IX	R_2TeCl_2 (HL) (4-methoxyphenyl)	$C_{28}H_{24}Cl_2N_2O_3Te$ (634.83)	Light Yellow (86)	198-200	52.65 (52.98)	3.82 (3.78)	4.30 (4.41)	20.25 (20.10)	11.02 (11.17)	16.56
X	$R_2Te(L)_2$ (4-methoxyphenyl)	$C_{42}H_{32}N_4O_4Te$ (784.09)	Orange yellow (82)	205-207	64.25 (64.34)	4.18 (4.08)	7.01 (7.15)	16.43 (16.27)	-	63.77
XI	R_2TeCl_2 (HL) (4-hydroxyphenyl)	$C_{26}H_{20}Cl_2N_2O_3Te$ (606.80)	Yellow (76)	184-186	51.32 (51.46)	3.22 (3.30)	4.50 (4.62)	21.25 (21.03)	11.52 (11.69)	8.24
XII	$R_2Te(L)_2$ (4-hydroxyphenyl)	$C_{40}H_{28}N_4O_4Te$ (756.06)	Light Orange (88)	190-192	63.35 (63.55)	3.84 (3.70)	7.25 (7.41)	16.62 (16.88)	-	57.77
XIII	R_2TeCl_2 (HL) (3-methyl-4-hydroxyphenyl)	$C_{28}H_{24}Cl_2N_2O_3Te$ (634.83)	Brown (81)	174-176	53.26 (52.96)	3.31 (3.81)	4.71 (4.41)	19.49 (20.09)	10.57 (11.17)	13.17
XIV	$R_2Te(L)_2$ (3-methyl-4-hydroxyphenyl)	$C_{42}H_{32}N_4O_4Te$ (784.09)	Reddish brown (85)	189-191	63.82 (64.32)	3.61 (4.11)	7.54 (7.14)	15.77 (16.27)	-	70.88

Values of Λ_M reported^{44, 45} for 1:1 electrolytes in DMSO = 50-70 $ohm^{-1} cm^2 mol^{-1}$

This conductance behavior of tellurium(IV) – Isatin Aniline Schiff base complexes is different from Sn(IV)¹⁵, Ti(IV) and Zr(IV)⁸ complexes, which are reported to be non-electrolytes in DMF.

Infrared spectra

The Infrared spectra of HIAN and its tellurium(IV) complexes are presented in Table 2. The spectra of Isatin-Aniline 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 parent ligand and RTeCl₃/R₂TeCl₂.

Table 2. Important IR Data (cm⁻¹) of Ligand (HIAN) and Complexes.

Compound No.	ν_{NH}	$\nu_{\text{C=O}}$	$\nu_{\text{C=N}}$	$\nu_{\text{C=N}^*}$	$\nu_{\text{C-O}}$
HIAN or HL	3165 sb	1739 s	1652 s	-	-
I	3194 s	1731 s	1614 s	-	-
II	-	-	1614 s	1580 m	1235 m
III	3160 mb	1730 s	1616 s	-	-
IV	-	-	1613 s	1592 s	1256 m, sh
V	3156 mb	1706 s	1614 s	-	-
VI	-	-	1617 s	1576 m, sh	1237 m
VII	3151mb	1731 s	1616 s	-	-
VIII	-	-	1617 s	1576 m	1237 m
IX	3152 mb	1706 s	1614 s	-	-
X	-	-	1614 s	1585 s	1249 s
XI	3170 sb	1720 sh	1614 s	-	-
XII	-	-	1614 s	1580 sh	1236 m
XIII	3171 mb	1727 sh	1615 s	-	-
XIV	-	-	1615 s	1580 sh	1235 m

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

The IR spectrum of the ligand (HIAN) shows strong bands at 3165, 1739 and 1652 cm⁻¹, which may be assigned¹⁵ to ν_{NH} , $\nu_{\text{C=O}}$ and $\nu_{\text{C=N}}$ of azomethine group respectively. In the case of TeCl₄(HL), RteCl₃(HL) and R₂TeCl₂(HL) complexes, $\nu_{\text{C=O}}$ of free HIAN at 1739 cm⁻¹ was shifted to lower regions between 1731-1706 cm⁻¹. This shift indicates^{8,15} the implication of carbonyl oxygen in the coordination at tellurium centre. The band appearing at 1652 cm⁻¹ in free HIAN, assignable to $\nu_{\text{C=N}}$ vibration mode, disappears in the spectra of complexes and strong band at around 1617 cm⁻¹ appears indicating participation of azomethinic nitrogen atom in the coordination. Also, the ν_{NH} band in HIAN at 3165(broad) remains unaffected in the complexes, showing its non participation the bonding with tellurium centre. Thus, the HL Schiff base ligand is coordinated to the tellurium atom as *NO* bidentate ligand.

In the infrared spectra of TeCl₂(L)₂, RteCl(L)₂ and R₂Te(L)₂, new IR bands are observed at 1576-1592 cm⁻¹ and 1235-1256 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^{8,15}. All these complexes exhibit strong band at around 1615 cm⁻¹ due to coordinated azomethinic nitrogen atoms. Thus, HIAN acts as a uninegative *NO* bidentate ligand in these 1:2 complexes. The broad band at 3165 cm⁻¹ in free ligand disappear in the complexes, there by exhibiting enolization of NH proton. 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.

¹H NMR spectra

Proton magnetic resonance spectra of aryltellurium(IV) Isatin-Aniline complexes are very complex and a lot of mixing of aryl proton signals of the HIAN and aryltellurium(IV) moiety takes place, thus making the independent assignment almost impossible. The chemical shift data for the free HIAN¹⁵ and Isatin-Aniline Schiff base complexes are compiled in Table 3.

Table 3. ¹H NMR spectral data of Schiff Base (HIAN) and complexes in DMSO-d₆

Compound No	Chemical Shift, δ ppm
HIAN or HL	6.39-6.92 (m, 5H, amine aromatic ring), 6.99-7.49 (m, 4H, isatin ring), 10.95 (s, 1H, NH)
I	6.91-7.58 (cm, 9H, Schiff base aryl protons), 11.046 (s, 1H, NH)
II	6.43-7.57 (cm, 18H, Schiff base aryl protons)
III	3.83 (s, 3H, -OCH ₃), 6.91-8.41 (cm, 13H, aryl protons of Schiff base & RTe), 11.024 (s, 1H, NH)
IV	3.36 (s, 3H, -OCH ₃), 6.39-8.19 (cm, 22H, aryl protons of Schiff base & RTe)
V	6.45-7.55 (cm, 13H, aryl protons of Schiff base & RTe), 8.16 (s, 1H, phenolic OH of RTe), 11.03 (s, 1H, NH)
VI	6.67-7.97 (cm, 22H, aryl protons of Schiff base & RTe), 8.25 (s, 1H, phenolic OH of RTe)
VII	2.17 (s, 3H, -CH ₃), 6.84-8.16 (cm, 12H, aryl protons of Schiff base & RTe), 10.02 (bs, 1H, phenolic OH of RTe), 11.045 (s, 1H, NH)
VIII	2.53 (s, 3H, -CH ₃), 6.69-8.27 (cm, 21H, aryl protons of Schiff Base & RTe), 10.11 (bs, 1H, phenolic OH of RTe)
IX	3.83 (s, 6H, -OCH ₃), 6.67-7.91 (cm, 17H, aryl protons of Schiff base and R ₂ Te), 11.047 (s, 1H, NH)
X	3.82 (s, 6H, -OCH ₃), 6.38-8.20 (cm, 26H, aryl protons of Schiff base and R ₂ Te)
XI	6.37-7.59 (cm, 17H, aryl protons of Schiff base and R ₂ Te), 8.24 (s, 2H, phenolic OH of R ₂ Te), 11.033 (s, 1H, NH)
XII	6.42-7.57 (cm, 26H, aryl protons of Schiff base and R ₂ Te), 8.16 (s, 2H, phenolic OH of R ₂ Te)
XIII	2.52 (s, 6H, -CH ₃), 6.20-7.42 (cm, 15H, aryl protons of Schiff base and R ₂ Te), 8.21 (s, 2H, phenolic OH of R ₂ Te), 11.041 (s, 1H, NH)
XIV	2.53 (s, 6H, -CH ₃), 6.41-7.57 (cm, 24H, aryl protons of Schiff base and R ₂ Te), 10.61 (s, 2H, phenolic OH of R ₂ Te)

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

Free HIAN shows two multiplets at 6.39-6.92 δ ppm and 6.99-7.49 δ ppm corresponding to amine aromatic ring and isatin skeleton, respectively and one singlet at 10.95 δ ppm corresponding to Isatin NH residue.

The proton NMR spectra of 1:1 complexes *i.e.* TeCl₄(HL), RTeCl₃(HL) and R₂TeCl₂(HL) display a downfield shift from 10.95 to 11.02-11.05 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^{15,46,47}. 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.* $\text{TeCl}_2(\text{L})_2$, $\text{RTeCl}(\text{L})_2$ and $\text{R}_2\text{Te}(\text{L})_2$ 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 HIAN 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 proposed (Figure 1) below:

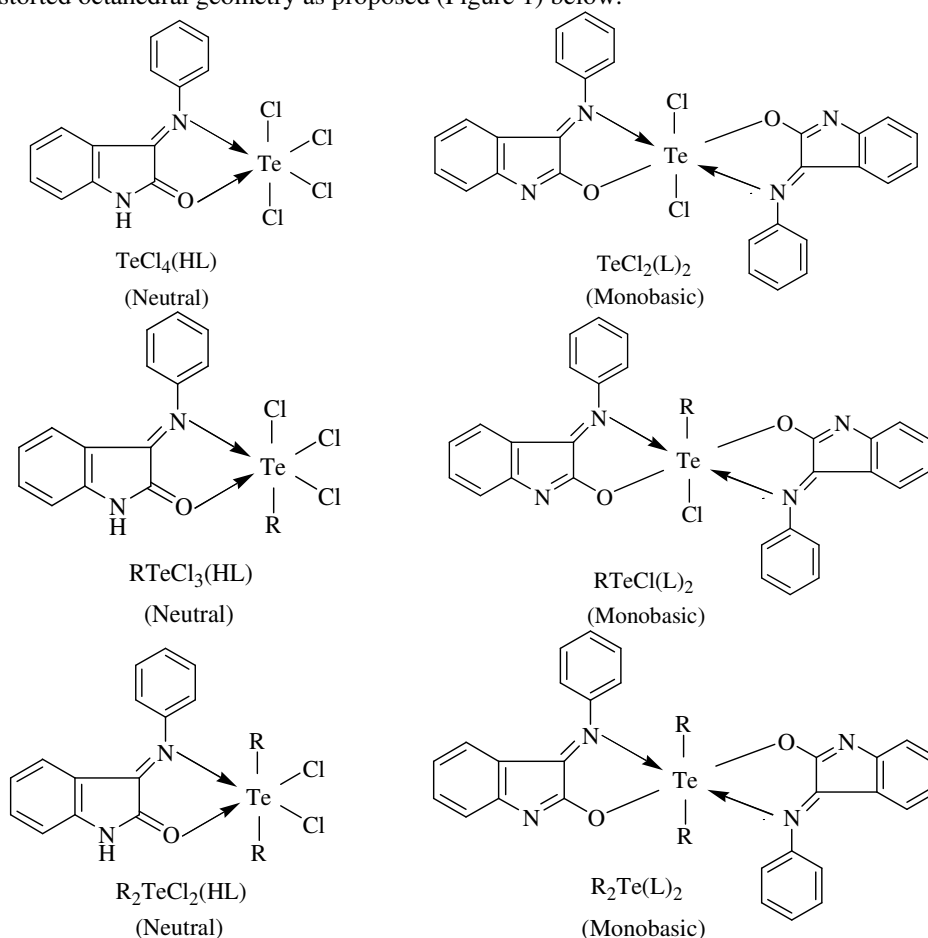


Figure 1. Proposed structures of tellurium(IV) Isatin-Aniline Schiff base complexes

Conclusion

The synthesis and characterization of new complexes of tellurium(IV) derivatives with a Schiff base derived from isatin and aniline (HL or HIAN) are reported. The synthesis were performed in conditions allowing the neutral bidentate and monobasic bidentate functions of the ligand to obtain the complexes of two types $\text{TeCl}_4(\text{HL})$, $\text{RteCl}_3(\text{HL})$, $\text{R}_2\text{TeCl}_2(\text{HL})$ and $\text{TeCl}_2(\text{L})_2$, $\text{RteCl}(\text{L})_2$, $\text{R}_2\text{Te}(\text{L})_2$. The synthesized complexes were characterized by elemental analyses, conductance measurement, IR and ^1H NMR spectral studies. Based on these studies, distorted octahedral stereochemistry has been assigned to these complexes.

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