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Studies on nickel (II) and palladium (II) complexes with some tetraazamacrocycles containing tellurium

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Abstract: The synthesis of 10-membered and 12-membered tellurium containing tetraazamacrocyclic complexes of divalent nickel and palladium by template condensation of diaryltellurium dichlorides, (aryl = p-hydroxyphenyl, 3-methyl-4-hydroxyphenyl, *p*-methoxyphenyl) with 1,2-diaminoethane and 1,3-diaminopropane in the presence of metal dichloride is reported. The resulting complexes have been subjected to elemental analyses, magnetic measurements, electronic absorption, infra-red, and proton magnetic resonance spectral studies. The formation of proposed macrocyclic skeletons and their donor sites have been identified on the basis of spectral studies. Distorted octahedral structure for the nickel complexes have been suggested.

Keywords: tetraazamacrocycles; diaryltellurium dichlorides; template condensation; ethylenediamine; 1,3-diaminopropane.

INTRODUCTION

The coordination chemistry of organotellurium ligands containing *hard* donor atoms such as nitrogen and oxygen along with *soft* tellurium is interesting as such ligand framework can provide 'insight' into competitive coordination behavior between the *hard* and *soft* donors towards a metal center.^{1,2} Such molecular systems may be important in transition metal catalyzed asymmetric synthesis,^{3,4} and as single source precursors in MOCVD processes.⁵⁻⁷ Also, macrocycle complexes are significant as they have been found to act as possible models for biochemically important proteins and enzymes.⁸⁻¹² Some recent publications¹³⁻¹⁵ exhibit the development of tellurium

Some recent publications¹³⁻¹³ exhibit the development of tellurium containing macrocycles. Srivastava *et al*¹⁶ reported the route of synthesis of metal complexes with tellurium containing macrocycles. In continuation of our earlier work,^{17,18} we herein report the synthesis and characterization of divalent nickel

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and palladium complexes with six novel tellurium tetraazamacrocycles (Te $_2N_4$ system).

RESULTS AND DISCUSSION

The formation of diaryltellurium(IV) dichlorides by the reactions of TeCl_4 with phenol,¹⁹ *o*-cresol²⁰ and anisole²¹ involves two step reactions. In first step, there is an electrophilic substitution of the phenyl ring by a trichlorotellurium moiety at a position *para* to the hydroxyl or the methoxy groups. This can be represented by the following equation :

$$R-H + TeCl_4 \rightarrow RTeCl_3 + HCl$$
(1)

(R = p-hydroxy, p-methoxy or 3-methyl-4-hydroxyphenyl).

These aryltellurium trichlorides, further react with phenol/*o*-cresol or anisole to give the diaryltellurium(IV) dichlorides as per equation below:

$$RTeCl_3 + R-H \rightarrow R_2TeCl_2 + HCl$$
(2)

These diaryltellurium dichlorides when refluxed with 1, 2-diaminoethane or 1, 3-diaminopropane in presence of $NiCl_2/PdCl_2$ in 2: 2:1 molar ratios yield the 10-membered and 12-membered tetraazamacrocyclic complexes respectively, as shown in Scheme - 1.

These complexes are colored, crystalline solids, fairly stable in dry air, and are soluble only in polar donor organic solvents.

Infra-red spectra

The important IR bands and their assignments are reported in Table I. The spectra are quite complex and an attempt has thus been made to draw the conclusions by comparing the spectra of metal complexes with those of corresponding constituent diarytellurium dichlorides and diaminoalkanes.

The metal complexes under study did not show bands characteristics of free NH₂ group, instead the entire complexes exhibit a single sharp absorption band at around 3180-3250 cm⁻¹ (sometimes mixed with O-H) attributed to V_{N-H} vibrations. The assignment of this sharp band is based on the fact that macrocyclic ligands which have coordinated secondary amino group, have bands^{18,22-24} in the vicinity of 3200 cm⁻¹. This contention finds support²² from appearance of bands of medium to strong intensity at 1627-1655 cm⁻¹ and 809-827 cm⁻¹ assigned as N-H deformation coupled with N-H out of the plane bending vibrations. Bands at 1156-1185 cm⁻¹ may be reasonably assigned to C-N stretching vibration.^{18,25,26} The above observation strongly suggests^{18,22,25,26} that proposed macrocyclic ring is supported by appearance of new weak intensity bands around 420-410 cm⁻¹ due to Te-N.^{18,27} Evidence for the formation of proposed macrocycles and coordination through N atoms is further supported by new medium to weak intensity bands around 480-450 cm⁻¹ assignable to Ni-N

stretching.²⁸ The M-Cl and Pd-N vibrations could not be ascertained due to non-availability of the far-infrared data.

Proton magnetic resonance spectra

The proton chemical shifts for metal complexes with 10-membered and 12membered tetraazamacrocycles which are soluble in DMSO- d_6 are presented in Tables II and III, respectively.

The phenyl protons in metal complexes resonate at slightly upfield side (6.89-7.88 δ ppm) as compared to parent diaryltellurium dichlorides^{19,20,29} due to an increase in electron density at the tellugium atom as a result of replacement of 2 Cl by 2 N atoms. Ethylenediamine, $H_2N-(CH_2)_2 - NH_2$ shows³⁰ two sets of four equivalent protons each at (a) = 1.19 δ ppm and (b) = 2.74 δ ppm .Metal complexes do not show any signal attributable to free – NH₂, instead a broad singlet at around 1.74-2.04 δ ppm, which may be assigned to coordinated secondary amino group³¹ is observed. This confirms the formation of proposed 10-membered macrocycle skeleton. The deshielding of –NH– protons further suggests the donation of electron density to the metal-ions. The methylene protons in these metal complexes resonate at 2.17-2.50 δ ppm, as a multiplet as reported^{18,24,26} for other tetraazamacrocyles derived from ethylenediamine.

1,3-Diaminopropane, NH₂-CH₂-CH₂-CH₂-NH₂, exhibits³² protons resonances at 1.15 (4H), 2.76 (4H) and 1.59 (2H) δ ppm due to amino, methylene (adjacent to N) and middle methylene groups, respectively. The metal complexes do not show any signal due to free amino group. Instead, a broad singlet at 1.78-1.91 δ ppm assignable to coordinated secondary amino group³¹ tellurium confirms formation 12-membered containing the of tetraazamacrocycles skeleton. The middle methylene protons and those adjacent to N-atoms resonate at 2.01 -2.50 and 2.86 -3.33 δ ppm, respectively. This behavior of complexes under study is quite similar to those of other tetraazamacrocycles^{18,24,33} derived from 1, 3-diaminopropane.

Further, the independence of aryl proton chemical shifts on the nature of metal- ions precludes the possibility of Te-M bond. The proton magnetic resonance studies on these Ni(II) and Pd(II) complexes support the tetra dentate nature of these ligands through four N-atoms as predicted by infrared studies.

Electronic absorption spectra and magnetic studies

The electronic absorption and magnetic moment data for the complexes are presented in Table IV.

The electronic spectral data of all the six Ni(II) complexes exhibit three spin allowed transitions from ${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}$, ${}^{3}T_{1g}(F)$, and ${}^{3}T_{1g}(P)$ which appear at 9850-10700 cm⁻¹, 13600-15052 cm⁻¹ and 24096-28668 cm⁻¹, respectively. This spectral pattern corresponds to a octahedral/distorted octahedral geometry^{16,34} Also, the

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ratio of $v_2 / v_1 \sim 1.4$ is an indicative^{36,37} of an octahedral stereochemistry for all these Ni(II) complexes. The third spin allowed d-d transitions appears as broad shoulder to the CT bands and extends up to 450 nm as reported³⁸ for other tetraazamacrocyclic complexes of Ni(II). The magnetic moment values in present Ni(II) complexes (2.90 - 3.44 μ_B) also suggest an octahedral stereochemistry for these complexes and rules out the possibility of square planer geometry.^{39,40} The Pd(II) complexes under study display two bands at 20829-24752 cm⁻¹ and 26900-28800 cm⁻¹ which may be assigned to ${}^{1}A_{1g} \rightarrow {}^{1}A_{2g}$, and ${}^{1}A_{1g} \rightarrow {}^{1}B_{1g}$. These transitions in Pd (II) complexes establish square planer coordination around palladium.^{34,41} This is further supported by their diamagnetic nature.

On the basis of above studies, nickel appears to be hexa coordinated especially in the solid state, presumably in a distorted octahedral fashion involving four N atoms of tetraazamacrocycles and two chlorine atoms, whereas palladium is tetra coordinated in a square- planar arrangement involving four N atoms of the macrocyclic ring. However, the proton magnetic resonance spectral pattern of nickel complexes also indicates⁴² the presence of diamagnetic square-planar configuration. Probably in solution, dissociation of chloride anions takes place and nickel (II) complexes form equilibrium of octahedral (paramagnetic) and square-planar (diamagnetic) species.

EXPERIMENTAL

All preparations were carried out under an atmosphere of dry N_2 and the solvents were dried and purified by the standard methods before use.

Preparation of diaryltellurium dichlorides

The bis(*p*-hydroxyphenyl), bis(3-methyl-4hydroxyphenyl), and bis(*p*-methoxyphenyl) tellurium dichlorides were prepared by the rections of tellurium tetrachloride with phenol, *o*-cresol, and anisole, respectively.¹⁹⁻²¹

Synthesis of metal complexes with tellurium containing 10-membered and 12-membered tetraazamacrocycles

The complexes were prepared by template condensation of diaryltellurium dichlorides with diaminoalkanes in presence of metal dichlorides in 2: 2: 1 molar ratio. A general procedure is given below.

A saturated methanolic solution of 4.0 mmol of diaryltellurium dichloride [1.538 g, 1.650 g, 1.650 g for bis(*p*-hydroxyphenyl), bis(3-methyl-4hydroxyphenyl) and bis(*p*-methoxyphenyl) tellurium dichlorides, respectively] was added to ethylenediamine (0.240 g, 4.0 mmol) or 1,3-diaminopropane (0.296 g, 4.0 mmol) in about 10 mL dry methanol with constant stirring. An immediate change in color was observed along with a little turbidity. The contents were stirred and refluxed for about 3 h. This was followed by addition of a solution of 2.0 mmol of metal dichloride (0.575 g, 0.355 g for NiCl₂.6H₂O and PdCl₂, respectively) in about 10 mL methanol. This resulted in a distinct change in color along with slight precipitation of a solid product. The solution was then refluxed for about 6 h and then cooled. A small amount of colored solid thus separated was filtered and the filtrate was concentrated to about one third of its original volume and kept in a freeze overnight to get the second crop

of the crystalline product. This was filtered and washed with benzene and dried in a vacuum desiccator over P_4O_{10} .

The purity of these compounds was checked by T.L.C using silica gel – G. The analytical data and physical properties of the complexes are presented in Table V.

Analytical methods and physical measurements

Carbon, hydrogen, and nitrogen analyses were obtained micro analytically from Sophisticated Analytical Instrumentation Facility, Panjab University, Chandigarh. The tellurium and chlorine contents were determined volumetrically⁴³ and palladium gravimetrically.⁴³ Nickel was estimated Atomic Absorption Spectrophotometerically. IR spectra were recorded in the region 4000-400 cm⁻¹ at SAIF, Panjab University, Chandigarh on a Perkin Elmer Model 2000 FTIR Spectrometer using the KBr pellets technique. ¹H NMR spectra were recorded at Kurukshetra University, Kurukshetra on BRUKER XWIN – NMR AVANCE 300 operating at 300.13 MHz in DMSO – d₆ using tetramethylsilane as an internal reference. The magnetic susceptibility data were obtained from National Physical Laboratory, New Delhi on a Gouy's balance (Model Johnson Matthey Alfa Products) using glycerin as a caliberant. The electronic spectra were recorded in dimethylsulphoxide on a Thermo Spectronic UV-1 UV-VISIBLE Spectrophotometer (Electron Corporation, India).

CONCLUSIONS

Nickel (II) and palladium (II) complexes have been prepared by condensation of diaryltellurium dichlorides with diaminoalkanes in the presence of metal chlorides. These metal complexes have been characterized using elemental analyses, magnetic measurements, electronic absorption, infra-red and proton magnetic resonance spectral techniques. A distorted octahedral structure for the Ni (II) complexes in the solid state and a square- planer structure for the Pd (II) complexes have been assigned on the basis of these studies. The Ni (II) complexes in solution probably form equilibrium of octahedral and square-planar species.

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ИЗВОД

ИСПИТИВАЊЕ КОМПЛЕКСА НИКЛА(II) И ПАЛАДИЈУМА(II) СА НЕКИМ ТЕРААЗАМАКРОЦИКЛИЧНИМ ЛИГАНДИМА КОЈИ САДРЖЕ ТЕЛУР

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Темплаттном кондензационом реакцијом полазећи од диарилтелур-дихлорида (арил = p-парахидроксифенил, 3-метил-4-хидроксифенил, p-метоксифенил) и 1,2-диаминоетана и 1,3-диаминопропана у присуству одговарајућег хлорида метала (Ni(II) и Pd(II)) синтетизовани су комплекси никла(II) и паладијума(II) са десеточланим и дванаесточланим тетраазамакроцикличним лигандима који садрже телур. Синтетисани комплекси су окарактерисани на основу резултата елементалне микроанализе, магнетних мерења, UV–Vis, IR и ¹H-NMR спектара. Грађење макроцикличног скелета са одговарајућим донорским атомима је праћено на основу спектроскопских мерења. На основу ових резултата је нађено

да комплекси никла(II) имају дисторговану окатедарску, док комплекси паладијума(II) имају квадратно-планарну геометрију.

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TABLES CAPTIONS

TABLE I. . Important IR data (cm⁻¹) for metal complexes

TABLE II. ^1H NMR data ($\delta\,\text{ppm})$ for 10-membered macrocyclic metal complexes in DMSO–d_6

TABLE III. ¹H NMR data (δ ppm) for 12-membered macrocyclic metal complexes in DMSO –d₆

TABLE IV. Electronic absorption spectra and magnetic moment data for metal complexes

TABLE V. Physical characteristics and elemental analysis of metal complexes

Complex	$\nu_{\rm N-H}$	N-H def.	N-H out of plane bending	$\nu_{\text{C-N}}$
$[NiL^1Cl_2]$	3180 m	1630 m	825 s	1185 m
$[NiL^2Cl_2]$	3191 m	1640 m	812 s	1176 m
[NiL ³ Cl ₂]	3210 m	1649 m	819 vs	1176 vs
$[NiL^4Cl_2]$	3250 m	1635 m	827 s	1173 s
[NiL ⁵ Cl ₂]	3250 m	1630 m	⁰ 813 s	1178 m
[NiL ⁶ Cl ₂]	3215 m	1645 m	822 s	1176 vs
$[PdL^1]Cl_2$	3210 m	1640 m	824 vs	1172 s
$[PdL^2]Cl_2$	3180 m	1635 m	809 vs	1173 m
$[PdL^3]Cl_2$	3230 m	1650 m	816 s	1176 s
$[PdL^4]Cl_2$	3190 s	1630 m	825 m	1156 vs
$[PdL^5]Cl_2$	3210 m	1627 m	809 s	1172 m
[PdL ⁶]Cl ₂	3210 m	1655 m	822 s	1177 vs

TABLE I. Important IR data (cm⁻¹) for metal complexes

*mixed with moisture band, s = strong, m = medium, vs = very strong.

TABLE II. ¹H NMR data (δ ppm) for 10-membered macrocyclic metal complexes in DMSO – d₆

Complex	-NH-	-CH ₂ -	-OCH ₃	Phenyl	–ОН		
[NiL ¹ Cl ₂]	1.45 ^s (4H)	2.17 ^m (8H)	_	$6.93^{d} (J = 8.7 Hz, 8H)$ $7.83^{d} (J = 8.7 Hz, 8H)$	9.85 ^b (4H)		
[PdL ¹]Cl ₂	1.40 ^s (4H)	2.50 ^m (8H)	_		8.35 ^b (4H)		
[PdL ³]Cl ₂	1.75 ^s (4H)	2.50 ^m (8H)	3.78 ^s (12H)	7.11 ^d $(J = 8.4 \text{ Hz})$,8H) 7.87 ^d $(J = 8.4 \text{ Hz})$ 8H)	_		
s = singlet, d = doublet, m = multiplet, b = broad							

TABLE III. ¹H NMR data (δ ppm) for 12-membered macrocyclic metal complexes in DMSO – d₆

Complex	-NH-	-CH ₂ - (middle)	-CH ₂ - (adjacent to N)	$-CH_3^*$	-OCH ₃	Phenyl	–OH
[NiL ⁴ Cl ₂]	1.89 ^s (4H)	2.27 ^m (4H) ^{2.86^m (8H)}	_	_	6.89^{d} (J = 8.7 Hz,8H) 7.77 ^d (J = 8.7 Hz,8H)	8.63 ^b (4H)
[PdL ⁴]Cl ₂	1.91 ^s (4H)	2.50 ^m (4H)*	2.88 ^m (8H)	-	-	6.92^{d} ($J = 8.7$ Hz,8H) 7.74^{d} ($J = 8.7$ Hz,8H)	8.14 ^b (4H)
[NiL ⁵ Cl ₂]	1.91 ^s (4H)	2.06 ^m (4H)	2.90 ^m (8H)	2.49 ^s (12H)	_	6.92^{d} (J = 8.4 Hz,4H) 7.34^{s} , 7.64 ^d (J = 8.4 Hz,8H)	8.10 ^b (4H)
[NiL ⁶ Cl ₂]	1.78 ^s (4H)	2.01 ^m (4H)	3.33 ^m (8H)	_	3.72 ^s (12H)	$6.89^{d} (J = 8.7)$ Hz, 8H) $7.88^{d} (J = 8.7)$ Hz, 8H)	_

* May be mixed with solvent peak, s = singlet, d = doublet, m = multiplet, b = broad.

	Absorption, cm ⁻¹				Observed Magnetic
Nickel(II)					Moment
Complex	$\boxed{\left[{}^{3}A_{2g} \rightarrow {}^{3}T_{2g}\right]}$	$[^{3}A_{2g} \rightarrow]$	${}^{3}T_{1g}(F)$	$\left[{}^{3}A_{2g} \rightarrow {}^{3}T_{1g}(P) \right]$	$\mu \mathrm{B}$
[NiL ¹ Cl ₂]	9994	14500		29740	3.44
[NiL ² Cl ₂]	9850	14040		24096	2.99
[NiL ³ Cl ₂]	10100	13600		25700	3.19
[NiL ⁴ Cl ₂]	10700	14525		28668	3.10
[NiL ⁵ Cl ₂]	10428	15052		27689	3.00
[NiL ⁶ Cl ₂]	9941	13855		26550	2.90
Palladium(II) Complex	$^{1}A_{1g} \rightarrow \ ^{1}A_{2g}$		$^{1}A_{1g} \rightarrow$	\rightarrow ¹ B _{1g}	
$[PdL^1]Cl_2$	24500*		27737		Diamagnetic
$[PdL^2]Cl_2$	21700		28490		Diamagnetic
[PdL ³]Cl ₂	24752*		26815		Diamagnetic
[PdL ⁴]Cl2	22026		27600		Diamagnetic
[PdL ⁵]Cl ₂	20829		28800		Diamagnetic
$[PdL^6]Cl_2$	21100		26900		Diamagnetic

TABLE IV. Electronic absorption spectra and magnetic moment data for metal complexes

 $^{1}A_{1g} \rightarrow {}^{1}E_{g}$

	Empirical formula (Formula weight)		mn		Elemental analyses found (calculated) %					
Complex		Color	°C (dec.)	Yield (%)	С	Н	N	Cl	Те	М
[NiL ¹ Cl ₂]	C ₂₈ H ₃₂ Cl ₂ N ₄ O ₄ Te ₂ Ni (873.4)	Pink	85-88	70	38.15 (38.51)	3.37)(3.69)	6.11 (6.41)	8.15 (8.12)	28.81 (29.22)	6.45 (6.51)
[NiL ² Cl ₂]	C ₃₂ H ₄₀ Cl ₂ N ₄ O ₄ Te ₂ Ni (929.5)	Red brown	145- 147	81	40.95 (41.35	3.87)(4.34)	6.11 (6.03)	7.61	26.71 (27.46)	6.18 (6.31)
[NiL ³ Cl ₂]	C ₃₂ H ₄₀ Cl ₂ N ₄ O ₄ Te ₂ Ni (929.5)	Light brown	157- 160	68	41.30 (41.35	4.44)(4.34)	6.26 (6.03)	7.59	26.97 (27.46)	6.06 (6.31)
[NiL ⁴ Cl ₂]	$C_{30}H_{36}Cl_2N_4O_4Te_2Ni$ (901.4)	Light orange	150- 152	70	39.52 (39.97	3.89)(4.03)	5.99 (6.22)	7.85 (7.87)	28.22 (28.31)	6.45 (6.51)
[NiL ⁵ Cl ₂]	$C_{34}H_{44}Cl_2N_4O_4Te_2Ni$ (957.5)	Red brown	135- 138	78	42.26 (42.65	4.33 (4.63)	5.37 (5.85)	7.43 (7.41)	26.10 (26.65)	6.04 (6.13)
[NiL ⁶ Cl ₂]	$C_{34}H_{44}Cl_2N_4O_4Te_2Ni$ (957.5)	Light green	177- 180	69	42.37 (42.65	4.41 (4.63)	5.68 (5.85)	7.27 (7.41)	27.19	5.88 (6.13)
[PdL ¹]Cl ₂	$C_{28}H_{32}Cl_2N_4O_4Te_2Pd$ (921.1)	Dark brown	155- 158	55	36.12 (36.51)	3.21)(3.50)	5.78 (6.08)	7.65 (7.70)	27.70 (27.71)	11.18 (11.55)
[PdL ²]Cl ₂	$C_{32}H_{40}Cl_2N_4O_4Te_2Pd$ (977.2)	Orange	210- 212	68	39.03 (39.33	3.93)(4.13)	5.24 (5.73)	7.38 (7.26)	26.40 (26.12)	10.62 (10.89)
[PdL ³]Cl ₂	$C_{32}H_{40}Cl_2N_4O_4Te_2Pd$ (977.2)	Orange	118- 120	50	39.16 (39.33	3.98)(4.13)	5.44 (5.73)	7.14 (7.26)	25.94 (26.12)	10.66 (10.89)
[PdL ⁴]Cl ₂	$C_{30}H_{36}Cl_2N_4O_4Te_2Pd$ (949.2)	Brown	168- 170	75	37.67 (37.96	3.53)(3.82)	5.46 (5.90)	7.47 (7.41)	26.09 (26.27)	10.83 (11.21)
[PdL ⁵]Cl ₂	$C_{34}H_{44}Cl_2N_4O_4Te_2Pd$ (1005.3)	Dark orange	222- 225	66	40.22 (40.62)	4.11)(4.41)	5.17 (5.57)	7.09 (7.05)	24.90 (25.39)	10.21 (10.59)
[PdL ⁶]Cl ₂	$C_{34}H_{44}Cl_2N_4O_4Te_2Pd$ (1005.3)	Orange	87-90	53	40.46 (40.62)	4.23)(4.41)	5.29 (5.57)	6.97 (7.05)	25.11 (25.39)	10.25 (10.59)

TABLE V. Physical characteristics and elemental analysis of metal complexes

SCHEME CAPTION

Scheme 1. Formation of Ni(II) and Pd(II) Complexes



- L^{3} L^{4} R = p-methoxyphenyl, n = 2R = p-hydroxyphenyl, n = 3
- L⁵; L⁶; R = 3-methyl-4-hydroxyphenyl, n = 3
- R = p-methoxyphenyl, n = 3

Scheme 1.

 L^1

 L^2

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