

Synthesis and Characterization of [(RR'Te)₂L] and [RR'Te.L] Compounds with Macrocyclic Ligands : A Photoelectron Spectroscopic Study

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ABSTRACT

[(RR'Te)₂.L¹], [RR'Te.L²] and [RR'Te.L³] compounds (where R = R' = C₆H₅CH₂; p-MeOC₆H₄; R = p-MeOC₆H₄ & R' = p-Me₂NC₆H₄; R = p-EtOC₆H₄ & R' = p-Me₂NC₆H₄; R = R' = C₆H₄C₆H₄) and L = L¹ = 3, 6, 9, 12, 20, 23, 26, 29, 35, 36-decaazatricyclo[29.3.1.1] hexatriaconta-1(34), 14, 16, 18(36), 31(35), 32-hexaene-2, 13, 19, 30-tetraone; L² = 3, 6, 9, 17, 20, 23, 29, 30- octaazatricyclo [23.3.1.1] triaconta-1 (28), 11, 13, 15(30), 25(29), 26-hexaene-2, 10, 16, 24-tetraone and L³ = 3, 7, 15, 19, 25, 26-hexaazatricyclo[19.3.1.1]hexacos-1(24), 9, 11, 13(26), 21(25), 22-hexaene-2, 8, 14, 20-tetraone have been synthesized and characterized by elemental analysis, molar conductance, I.R. and X-ray photoelectron spectra (XPS) data and an distorted octahedral geometry were established.

Keywords: Macrocyclic Ligands, Organotellurium (II), XPS.

I. INTRODUCTION

Macrocyclic ligands and their metal complexes have attracted extensive research interest in the past decades, due to its applications¹⁻⁹ and their importance in coordination chemistry¹⁰ and bioinorganic chemistry¹¹⁻¹³. Many macrocyclic ligands such as crown ethers¹⁴; porphyrins¹⁵; saturated and unsaturated polyamines¹⁶⁻²¹; polyazamacrocycles²²⁻²⁷; N₄S₂ donor macrocyclic²⁸⁻³⁶. Robson type tetraaminodiphenol macrocyclic ligands³⁷⁻⁴³ have been synthesized and characterized in last few decades. The studies on complexes of Schiff base macrocyclic ligands with different size, number and donor atoms for coordination with a variety of metal centres have been studied⁴⁴. Moreover, the template condensation reaction lies in the heart of macrocyclic chemistry⁴⁵⁻⁴⁷. Many transition metal ions are found in living systems in macrocyclic ligand environment and work as enzymes or carriers⁴⁸⁻⁵⁰.

Macrocyclic metal complexes have been used as detecting tumor lesions⁵¹; as in labeling monoclonal antibodies with radioactive models⁵²⁻⁵⁴; as metal ion separation⁵⁵⁻⁵⁷; as cancer diagnosis⁵⁸⁻⁶⁰; as

radiotherapeutic and therapeutic⁶¹⁻⁶²; as catalyst⁶³⁻⁶⁶; as pharmacological agents⁶⁷⁻⁶⁹; as biological process such as photosynthesis and dioxygen transport⁷⁰⁻⁷¹; as photosensitizer⁷²⁻⁷³; as versatile coordination behaviour⁷⁴⁻⁷⁵; as toxicity against bacterial fungal growth⁷⁶; as anticancerous⁷⁷; as antitumor⁷⁸; as environment importance⁷⁹; as potential medicinal applications such as magnetic resonance imaging (MRI)⁸⁰⁻⁸¹; as N.M.R. shift and relaxation agents⁸²⁻⁸³; as RNA cleavage catalyst⁸⁴⁻⁸⁵. The different type of macrocyclic ligands and their metal complexes discoveries have created supramolecular chemistry and its enormous diversity⁸⁶⁻⁹⁰. Very recently few comprehensive reviews on macrocyclic ligands and their metal complexes with their applications have been also appeared⁹¹⁻⁹⁶. Very recently the development of compartmental macrocyclic Schiff bases and related polyamine derivatives have been also published⁹⁷. Recently, the electron donor properties of organotellurium (IV) and (II) of macrocyclic compounds i.e. as Lewis base are well known⁹⁸⁻¹⁰⁷, but the electron acceptor i.e. as Lewis acid properties of organotellurium (II) of macrocyclic compounds are less known¹⁰⁸⁻¹¹². This paper deals with the acceptor

properties of organotellurium (II) macrocyclic compounds.

II. METHODS AND MATERIAL

Experimental

Te powder and TeCl_4 ; all chemicals and solvents were of A R Grade and purchased from Aldrich and BDH and were used after distillation and further purification. $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{Te}$; $(\text{C}_6\text{H}_4)_2\text{Te}$; $(p\text{-MeOC}_6\text{H}_4)_2\text{Te}$; $(p\text{-MeOC}_6\text{H}_4)$ $(p\text{-Me}_2\text{NC}_6\text{H}_4)\text{Te}$ and $(p\text{-EtOC}_6\text{H}_4)$ $(p\text{-Me}_2\text{NC}_6\text{H}_4)\text{Te}$ were prepared, purified and characterized as reported in literature¹¹³⁻¹¹⁷.

Preparation of Metal Compounds

A. Preparation of $[(\text{RR}'\text{Te})_2\text{L}^1]$ compounds with macrocyclic ligand (L^1) i.e. 3, 6, 9, 12, 20, 23, 26, 29, 35, 36 –decaazatricyclo [29.3.1.1] hexatriaconta-1 (34), 14, 16, 18(36), 31(35), 32-hexaene-2, 13, 19, 30- tetraone ligands :

Triethylenetetraamine (1 mmol) was mixed with $\text{RR}'\text{Te}$ (1 mmol) in dry ethanol and refluxed for 1 hr. and then put dropwise pyridine 2,6- dicarboxylic acid into this solution (2mmol : 2mmol ratio) and again refluxed this solution for 2 hrs. . The resulting precipitate was filtered and recrystallized into ether : ethanol (9: 1 ratio). (Fig. 01).

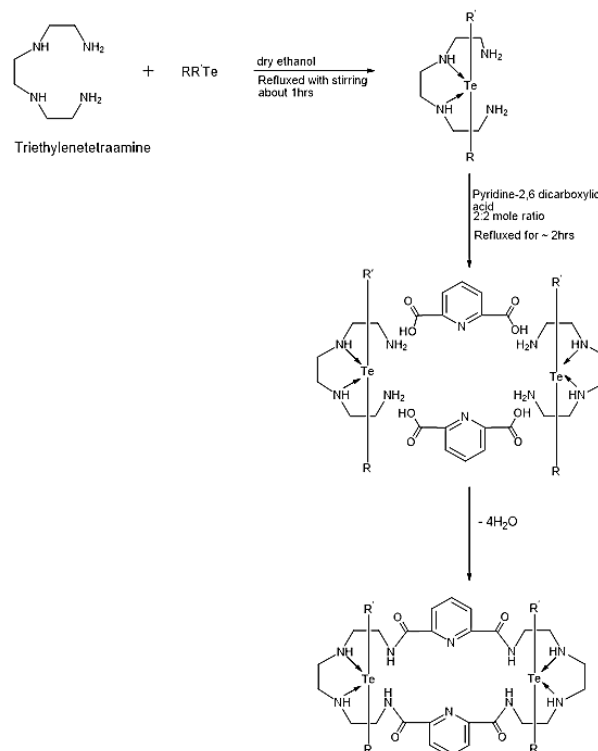


Figure 1. Proposed mechanism of $[(\text{RR}'\text{Te})_2\text{L}^1]$ compounds i.e. Homobimetallic compounds where $\text{L}^1 = 3, 6, 9, 12, 20, 23, 26, 29, 35, 36$ –decaazatricyclo [29.3.1.1]hexatriaconta-1(34), 14, 16, 18(36), 31(35), 32-hexaene-2, 13, 19, 30- tetraone.

B. The preparation of $[(\text{RR}'\text{Te}). \text{L}^2]$ compounds with macrocyclic ligand (L^2) i.e. 3, 6, 9, 17, 20, 23, 29, 30- octaazatricyclo [23.3.1.1] triaconta -1 (28), 11, 13, 15(30), 25(29), 26- hexaene -2, 10, 16, 24 –tetraone.

The diethylenetriamine (2mmol) was mixed with $[\text{RR}'\text{Te}]$ (1mmol) in dry ethanol and refluxed for 1 hr. and then pyridine- 2,6-dicarboxylic acid (2mmol :1mmol ratio) is mixed into above solution and refluxed for 2 hrs. . The resulting precipitate was filtered and recrystallized by benzene-pet-ether (9:1 ratio) solution (Fig. 02).

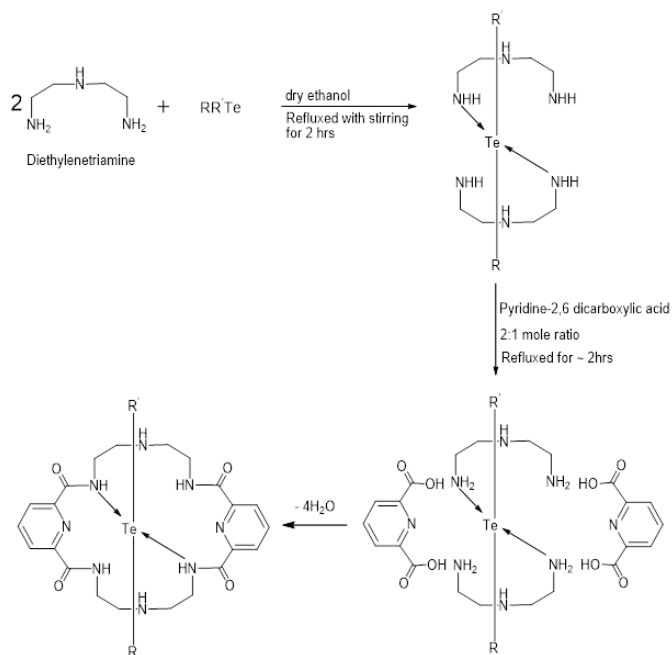


Figure 2. Proposed mechanism of $[(RR'Te)L^3]$ compounds i.e. Homomonometallic compounds where- $L^3 = 3, 7, 15, 19, 25, 26$ -hexaazatricyclo [19.3.1.1] hexacosa 1(24), 9, 11, 13(26), 21(25), 22 -hexaene - 2, 8, 14, 20 -tetraone.

C. Preparation of $[(RR'Te)L^3]$ compounds with macrocyclic ligand (L^3) i.e. 3, 7, 15, 19, 25, 26-hexaazatricyclo[19.3.1.1]hexacosa 1(24), 9, 11, 13(26), 21(25), 22 -hexaene - 2, 8, 14, 20 -tetraone

The 1,3 - diaminopropane (2mmol) was mixed with $[RR'Te]$ (1mmol) in dry ethanol and refluxed for 1 hr. and then pyridine 2,6- dicarboxylic acid (2mmol : 1mmol ratio) was mixed into the above solution and again refluxed for 2 hrs.. The resulting precipitate was filtered and recrystallized by benzene-pet-ether (9:1 ratio) solution (Fig. 03).

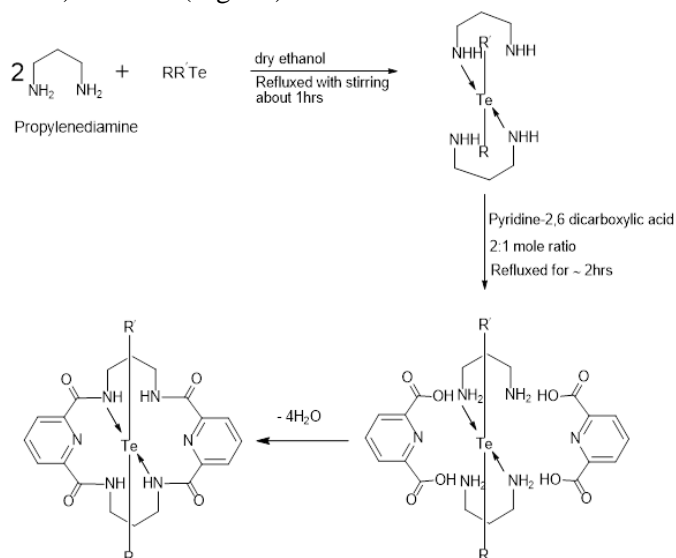


Figure 2. Proposed mechanism of $[(RR'Te)L^3]$ compounds i.e. Homomonometallic compounds where- (L^3)= 3, 7, 15, 19, 25, 26-hexaazatricyclo [19.3.1.1] hexacosa 1(24), 9, 11, 13(26), 21(25), 22 -hexaene - 2, 8, 14, 20 -tetraone.

Characterization of the Complexes

The elemental analysis for C, H, N and Te metal were determined on a semi micro scale at Central Drug Research Institute, Lucknow, U.P. India. Molar conductance of all the complexes were measured at room temperature in acetone by Digisum Electronic Conductivity Bridge. Infra-Red spectra of all these complexes were recorded in KBr and CsI on Perkin-Elmer 457 spectrometer. The X-ray photoelectron spectra i.e. XPS were recorded on a VG scientific ESCA-3 MK II electron spectrometer. The Mg $k\alpha$ X-ray line (1253.6 eV) was used for photo excitation. The Cu $2p_{3/2}$ (BE = 932.8 ± 0.2 eV) and Au $4f_{7/2}$ (BE = 83.8 ± 0.1 eV) lines were used to calibrate the instrument and Ag $3d_{5/2}$ (BE = 368.2 eV) was used for cross-checking. All the spectra were recorded using the same spectrometer parameter of 50 eV pass energy and 4mm slit width. The reduced full width at half maximum (FWHM) at the Au $4f_{7/2}$ (BE = 83.8 eV) level under these conditions was 1.2 eV. The powdered sample was mixed with high purity silver powder to reduce the charging effect. A thin layer of such a sample was pressed on a gold metal gauge which was welded to a nickel sample holder. The Ag $3d_{5/2}$ (BE = 368.2 eV) level obtained from this sample was sharp and did not show any observable shift. Thus the charging of the samples if present at all was negligible⁷. The spectra were recorded in triplicate in the region of the interest. In most of the cases the binding energies were reproducible within ± 0.1 eV. The usual least square fitting procedure of determining peak positions, line width and areas was used.

III. RESULTS AND DISCUSSION

All these newly synthesized compounds are soluble in common organic solvent and stable towards atmospheric oxygen and moisture. The element analysis for C, H, N and Te were found within range $\pm 0.5\%$. The molar conductance of 10^{-3} M solution of the compounds was determined at room temperature were observed in range from $20-30 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ in acetone and 0.82 to $1.02 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$ in nitrobenzene indicating absence of ionic species¹¹⁹. Both

elemental analysis and molar conductance suggested the composition of prepared compounds as $[(RR'Te)_2L^1]$; $[RR'Te.L^2]$ and $[RR'Te.L^3]$.

Infrared frequencies associated with fundamental modes of vibration of ν_{Te-C} and ν_{Te-N} was observed in the range $230-260\text{ cm}^{-1}$ and $410-420\text{ cm}^{-1}$.¹²⁰

The binding energies (eV) of $[(RR'Te)_2L^1]$; $[RR'Te.L^2]$ and $[RR'Te.L^3]$ for Te $3d_{3/2,5/2}$ and N1s photoelectron peaks are listed in table. 1 (Fig. 4 to 5). It was observed that the binding energies of Te $3d_{3/2,5/2}$ in the starting material $RR'Te$ was higher than their prepared molecular adducts $[(RR'Te)_2L^1]$; $[RR'Te.L^2]$ and $[RR'Te.L^3]$. These observations suggested that electron density on tellurium metal ion has increased due to coordination of ligand with tellurium metal ion¹¹⁸(Fig.4). Moreover, N1s photoelectron spectra in

$[(RR'Te)_2L^1]$ compounds have shown two N1s photoelectron with intensity ratio of 6:4 at binding energies ~ 401 & ~ 404 eV suggested that six nitrogen atoms are uncoordinated while four are coordinated. Similarly, in $[RR'Te.L^2]$ and $[RR'Te.L^3]$ N1s photoelectron spectra have shown two N1s photoelectron peak, one at ~ 401 and another at ~ 404 eV with 6:2 and 4:2 intensity ratio respectively, suggesting six nitrogen atoms are uncoordinated and two nitrogen atoms are coordinated in $[RR'Te.L^2]$ compounds while in $[RR'Te.L^3]$ four nitrogen atoms are uncoordinated and two nitrogen atoms are coordinated (Fig.5)¹¹⁸.

Thus elemental analysis, molar conductance, I.R. and X-ray photoelectron spectra (XPS) data suggested that all these molecular adducts are having distorted octahedral geometry.

Table 1: Te $3d_{3/2,5/2}$ and N1s binding energies (eV) in $[RR'Te]$ and $[(RR'Te)_2L^1]$, $[RR'Te.L^2]$, $[RR'Te.L^3]$ compounds

S. No.	Compounds	Te		N1s	
		$3d_{3/2}$	$3d_{5/2}$	Uncoordinated	Coordinated
1	$(C_6H_5)_2Te$	586.0	575.8	-	-
2	$[(C_6H_5)_2Te]_2.L^1$	584.0	573.2	401.4(6)	404.8(4)
3	$(C_6H_5)_2Te.L^2$	584.0	573.2	401.4(6)	404.8(2)
4	$(C_6H_5)_2Te.L^3$	584.0	573.2	401.4(4)	404.8(2)
5	$(p-MeOC_6H_4)_2Te$	586.2	575.6	-	-
6	$[(p-MeOC_6H_4)_2Te]_2.L^1$	584.2	573.2	401.4(6)	404.8(4)
7	$(p-MeOC_6H_4)_2Te.L^2$	584.2	573.2	401.4(6)	404.8(2)
8	$(p-MeOC_6H_4)_2Te.L^3$	584.2	573.2	401.4(4)	404.8(2)
9	$(p-MeOC_6H_4)(p-Me_2NC_6H_4)Te$	586.2	575.6	-	-
10	$[(p-MeOC_6H_4)(p-Me_2NC_6H_4)Te]_2.L^1$	584.0	573.2	401.4(6)	404.8(4)
11	$(p-MeOC_6H_4)(p-Me_2NC_6H_4)Te.L^2$	584.0	573.2	401.4(6)	404.8(2)
12	$(p-MeOC_6H_4)(p-Me_2NC_6H_4)Te.L^3$	584.0	573.2	401.4(4)	404.8(2)
13	$(p-EtOC_6H_4)(p-Me_2NC_6H_4)Te$	586.2	575.6	-	-
14	$[(p-EtOC_6H_4)(p-Me_2NC_6H_4)Te]_2.L^1$	584.0	573.2	401.4(6)	404.8(4)
15	$(p-EtOC_6H_4)(p-Me_2NC_6H_4)Te.L^2$	584.0	573.2	401.4(6)	404.8(2)
16	$(p-EtOC_6H_4)(p-Me_2NC_6H_4)Te.L^3$	584.0	573.2	401.4(4)	404.8(2)
17	$(C_6H_5CH_2)_2Te$	586.2	575.8	-	-
18	$[(C_6H_5CH_2)_2Te]_2.L^1$	584.0	573.2	401.4(6)	404.8(4)

19	$(C_6H_5CH_2)_2Te.L^2$	584.0	573.2	401.4(6)	404.8(2)
20	$(C_6H_5CH_2)_2Te.L^3$	584.0	573.2	401.4(4)	404.8(2)
21	$(C_6H_4C_6H_4)Te$	586.2	575.8	-	-
22	$[(C_6H_4C_6H_4)Te]_2.L^1$	584.0	573.2	401.4(6)	404.8(4)
23	$(C_6H_4C_6H_4)Te.L^2$	584.0	573.2	401.4(6)	404.8(2)
24	$(C_6H_4C_6H_4)Te.L^3$	584.0	573.2	401.4(4)	404.8(2)

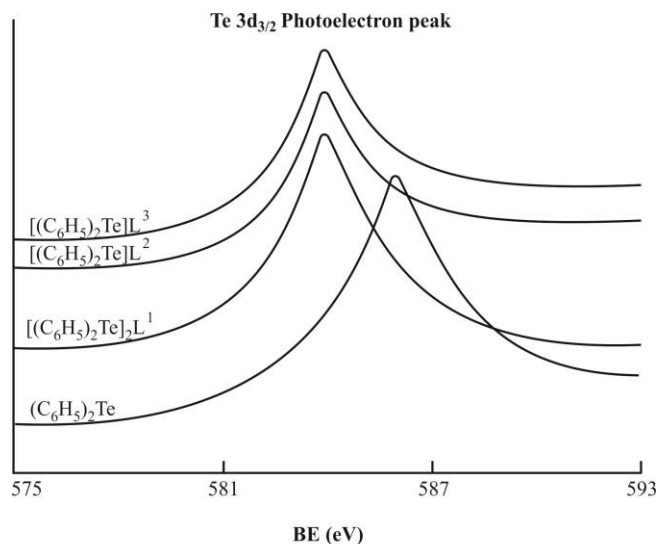


Fig 04 : Te 3d_{3/2} binding energies (eV) in $(C_6H_5)_2Te$; $[(C_6H_5)_2Te]L^1$, $[(C_6H_5)_2Te]L^2$ and $(C_6H_5)_2Te.L^3$ compounds

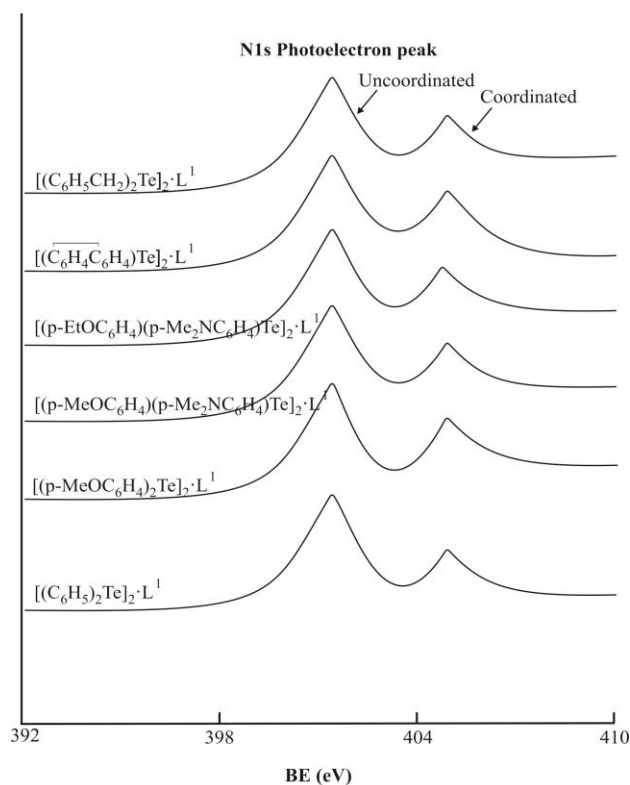


Figure 5. N1s binding energies (eV) in $[(RR'Te)_2].L^1$ compounds

IV. ACKNOWLEDGEMENT

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