



Synthesis and Characterization of Some New [RR'SeL] Complexes (L = Macrocyclic Ligand): A Photoelectron Spectroscopic Study (Part-II)

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Forty eight complexes of type [RR'SeL] (where R = R' = CH₃; R = R' = *n*-C₄H₉; R = R' = *n*-C₈H₁₇; R = R' = *i*-C₃H₇; R = R' = C₆H₅; R = C₆H₅, R' = C₂H₅; R = C₆H₅, R' = CH₃ and R = C₆H₅, R' = CH₂Cl; L = newly prepared macrocyclic ligands) have been synthesized and characterized by elemental analysis, molar conductivity, IR and XPS data. An octahedral geometry was established for all these prepared complexes.

Keywords: Selenium, Macrocyclic ligand, X-ray photoelectron spectroscopy.

INTRODUCTION

The chalcogenolates have attracted enormous attention owing to their importance in biological systems and their ability to form metal complexes and clusters of various bonding modes and structural motifs [1-7]. The recent interest in the study of metal chalcogenolate complexes stems from the potential use of these complexes in preparation of semiconducting metal selenides and tellurides [8] and nanoparticles [9]. Important application of alkali and alkaline earth metal chalcogenolates in synthetic and technical chemistry resulted in reports of the number of targeted molecules [10-12]. Metal chalcogenides (M_xE_y; M = Pd or Pt, E = S, Se or Te) find several applications in catalysis and the electronic industry. Metal chalcogenolates have an attractive potential to serve as precursors for the synthesis of such chalcogenides. These metal complexes are isolated as polymeric, non-volatile, insoluble or sparingly soluble. To suppress polymerization, sterically demanding chalcogenolate ligands have been employed. Organoselenolato metal complexes can be synthesized *via* selenium insertion into alkali metal-transition metal bonds of carbonylates and subsequent reactions with organohalides [13]. Corrigan and coworkers [14] have synthesized Zn(II) selenolate complexes and used it as capping agent in the synthesis of nanoclusters. Puddephat *et al.* [15] have

reported the first cationic Pt(VI) selenolate complexes by reacting adamantanoid compound [Hg₄(SePh)₆(L)₄][ClO₄]₂ with [PtMe₂(bu₂bpy)]. Laitine *et al.* [16] have prepared a series of metal selenolate complexes [Pt(SeR)₂(PPh₃)₂] which are significant as synthons for more complicated species. Many transition metal chalcogenolates *e.g.* Ni(II) [17], Pd(II), Pt(II) [18] and Zn(II) [19,20] selenolate complexes have been prepared with bulky ligands. Recently, Yam *et al.* have prepared a series of dinuclear copper(I) [21,22] and Ag(I)-chalcogenolate complexes [23] and studied their photophysics. The structure and bonding studies of transition-metal chalcogenolates are well documented, analogous studies with the more electropositive elements (*i.e.* alkali, alkaline earths and *f*-block elements) are less well defined. Structural studies of ionic chalcogenolates are generally hampered by thermal sensitivity and dissociation tendency in neutral donor solvents. The unstable lithium selenolate undergoes self-addition, which eventually yields uniformly a complex macrocyclic ring [24]. Magnesium selenolate showed selective Michael/aldol induction for preparation of anti-aldols from β-substituted α,β-unsaturated esters [25,26]. Much of the recent interest in lanthanide chalcogenolates results from their possible use as precursors to lanthanide chalcogenolates. These materials are of wide interest due to their unusual magnetic, electronic and optical properties [27]. A wide variety

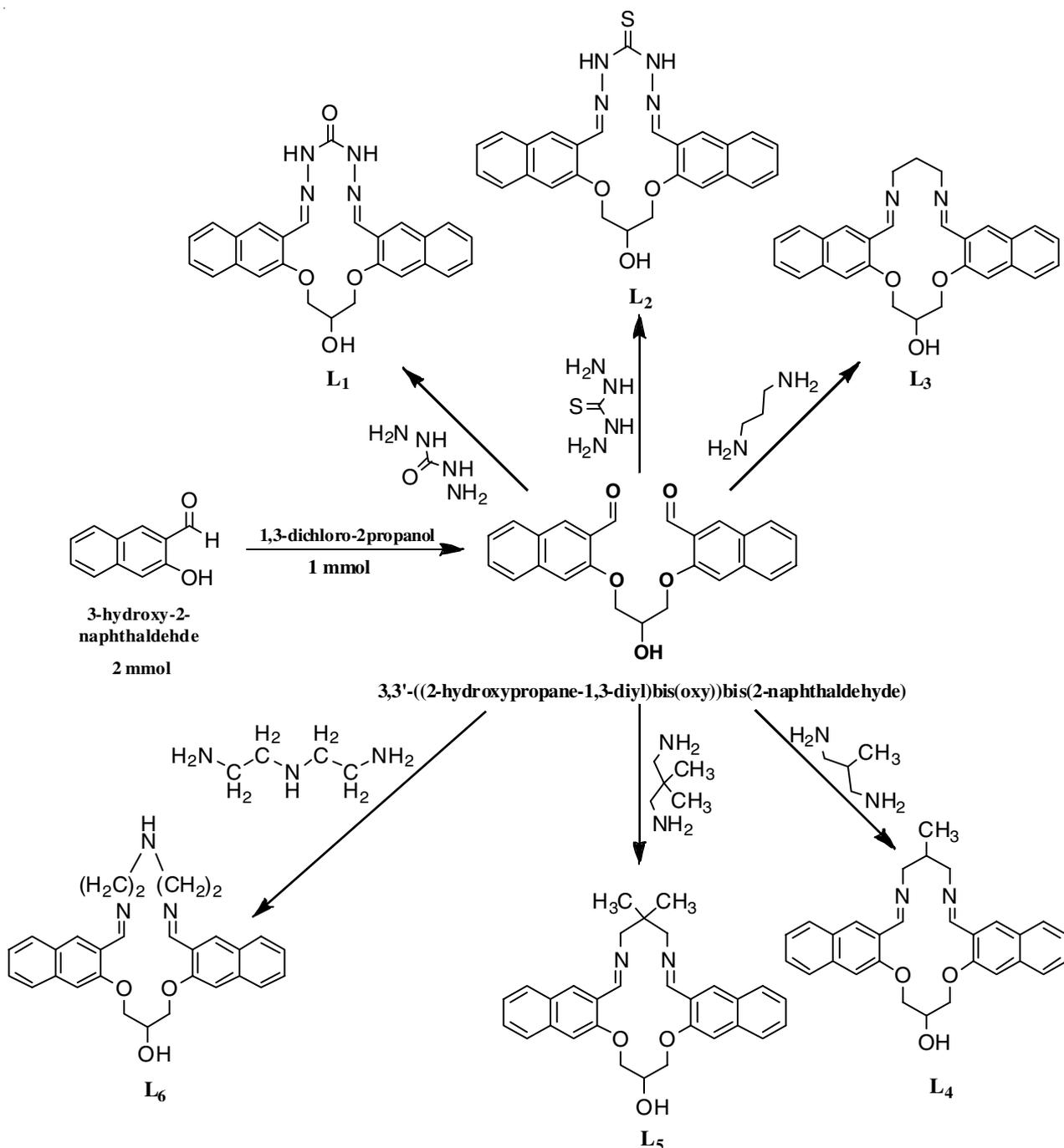
of chalcogenolate ligands, each imparting unique physical properties, have been used to stabilize divalent and trivalent lanthanide ions. Samarium selenolates ($RSeSmI_2$) have been prepared by reduction of diselenides by SmI_2 , which 3-substituted-1-chlorocarbonylimidazolidin-2-ones to give the corresponding selenoesters in good yields under mild and neutral conditions [28].

In this article, the complexation behaviour of diorgano-selenium(II) *i.e.* $RR'Se$ where $R = R' = CH_3$; $R = R' = n-C_4H_9$; $R = R' = i-C_3H_7$; $R = R' = n-C_8H_{17}$; $R = R' = C_6H_5$; $R = C_6H_5$ $R' = C_2H_5$; $R = C_6H_5$ $R' = CH_3$ and $R = C_6H_5$ $R' = CH_2Cl$ with various newly synthesized macrocyclic ligands *i.e.* (L_1 to L_6) in 1:1 molar ratio have reported.

EXPERIMENTAL

The elemental analysis for C, H and N were determined on a semimicro scale at CDRI, Lucknow, India. Molar conductances of all the complexes were measured at room temperature in acetone by Digisum Electronics Conductivity bridge. The infrared spectra of the complexes and ligands were recorded in KBr and CsI on Perkin-Elmer 457 spectrometer.

The X-ray photoelectron spectra (XPS) were recorded on a VG scientific ESCA-MK II electron spectrometer. The $MgK\alpha$ X-ray line (1253.6 eV) was used for photo-excitation. The $Cu\ 2P_{3/2}$ (BE = 932.8 0.2) and $Au\ 4f_{7/2}$ (BE = 83.8 0.1) lines were used for cross-checking the instrument and $Ag\ 3d_{5/2}$



(BE = 368.2) was used for cross-checking [29]. All the spectra were recorded using the same spectrometer parameter of 50 eV pass energy and 4 mm slit width. The reduced full width half maximum (FWHM) at 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 gold metal gauze which was welded to a nickel sample holder. The Ag 3d_{5/2} level (BE = 368.2eV) obtained from this sample was sharp and did not show any observable shift. Thus the charging of the sample if at all present was negligible [29].

Synthesis of ligands: RR'Se (where R = R' = CH₃; R = R' = n-C₄H₉; R = R' = i-C₃H₇; R = R' = n-C₈H₁₇; R = R' = C₆H₅; R = C₆H₅, R' = C₂H₅; R = C₆H₅, R' = CH₃ and R = C₆H₅, R' = CH₂Cl) have been synthesized according to the literature [30-32].

Synthesis of 2-[3-(formylphenoxy)-2-hydropropoxy]-naphthaldehyde: A solution of NaOH (8.0 g, 0.2 mol) in water (400 mL) was added to a ethanolic solution of naphthaldehyde (20.98 mL, 0.2 mol). The resulting solution was heated upto 55-60 °C 2h and then 1,3-dichloro-2-propane (9.54 mL, 0.1 mol) was added. The solution was made homogenous by addition of sufficient amount of ethanol. The resulting solution was refluxed for 4 h (pH 8-9) at 80 °C and then cooled at 0 °C. The cream coloured solid product was collected by filtration and dried in a vacuum over P₄O₁₀ (Scheme-I).

Synthesis of macrocyclic ligands: To a hot ethanolic solution (20 mL) of 2-[3-(formylphenoxy)-2-hydropropoxy] naphthaldehyde (6 g, 0.02 mol) a hot solution of NH₂NHCO-NHNH₂ or NH₂NHCSNHNH₂ or NH₂(CH₂)₃NH₂ or NH₂CH₂CH(CH₃)CH₂NH₂ or NH₂CH₂C(CH₃)₂CH₂NH₂ or NH₂(CH₂)₂NH(CH₂)₂NH₂ (0.02 mol) was added with constant stirring. The solution was heated at 50-60 °C (pH 8) for 3 h. On cooling at 0 °C, a solid product was precipitated out. It was filtered

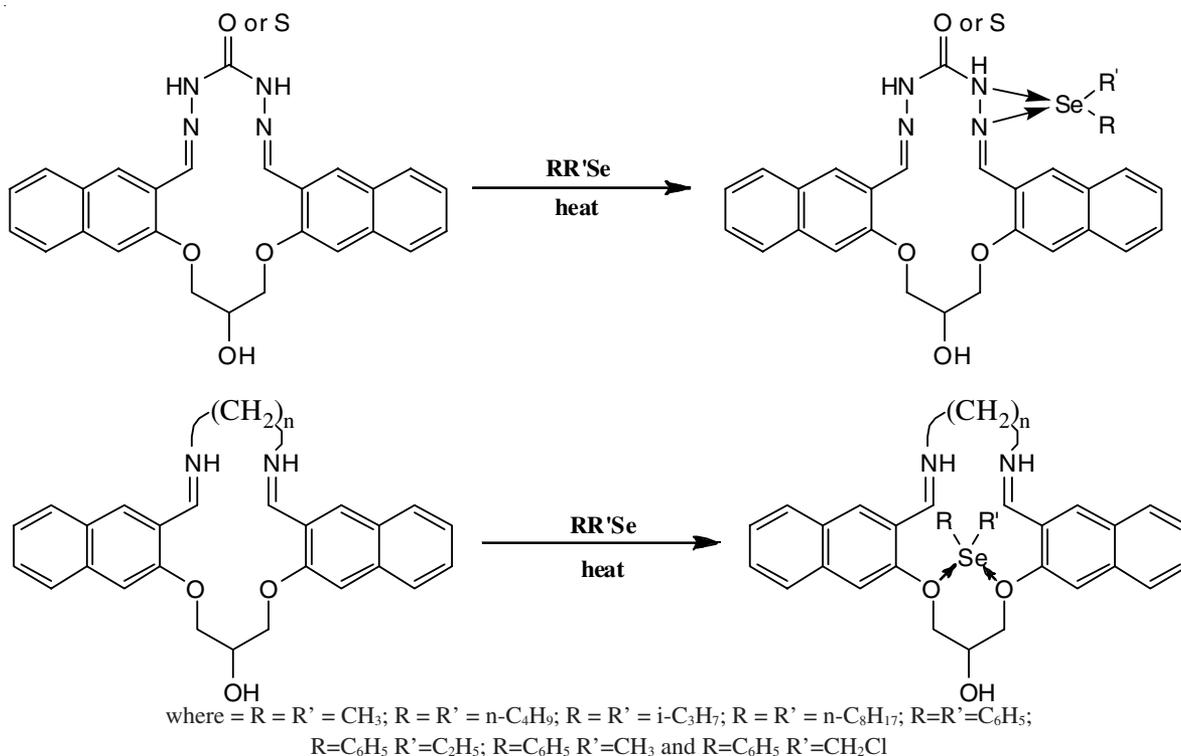
washed several times with cold ethanol and dried over P₄O₁₀ (Scheme-I).

Synthesis of metal complexes: Hot ethanolic solution of RR'Se (1 mmol) (20 mL) was added dropwise to magnetically stirred solution of macrocyclic ligand (1 mmol) in ethanol (30 mL) L₁ or L₂ or L₃ or L₄ or L₅ or L₆. The resulting mixture was refluxed for 3 h and then clear solution was allowed to cool at 0 °C. The solid product precipitated out which was filtered washed with ethanol, diethylether and dried under vacuum over P₄O₁₀ (Scheme-II).

RESULTS AND DISCUSSION

The molar conductance values were obtained in range from 20-30 Ω⁻¹ cm² mol⁻¹ in acetone suggesting non-electrolyte nature of all these complexes [33]. The elemental analysis values for these all prepared complexes for C, H, N were observed within ± 0.5 %.

In the IR spectra of ligand L₁ and L₂ and their respective [RR'SeL₁₋₂] complexes, ν(C=N) one component band appears at higher frequency ~1640-1610 cm⁻¹ and other at same position as in their respective ligand; suggesting one ν(C=N) group nitrogen is coordinated and other ν(C=N) is uncoordinated in these [RR'SeL₁₋₂] complexes [34,35]. Similarly, in IR spectra of ligand L₁ and L₂ and their respective [RR'SeL₁₋₂] complexes, ν(N-H) have not shown same frequency in the region 3300-3200 cm⁻¹. The ν(N-H) one component band appear at higher frequency ~ 3365-3220 cm⁻¹ and other on same frequency position, suggesting one nitrogen of ν(N-H) is coordinated and other is uncoordinated in these [RR'SeL₁₋₂] complexes [34,35]. But the IR spectra of ligands L₃ or L₄ or L₅ or L₆ and in their respective [RR'SeL₃₋₆] complexes, ν(C=N) have shown same frequency in the region 1620-1600 cm⁻¹, suggesting non-involvement of ν(C=N) in complexation [31,32]. Similarly in



Scheme-II

TABLE-1
Se 3p_{1/2}; N 1s, O 1s AND S 2p_{1/2,3/2} BINDING ENERGIES (eV) LIGAND (L) AND THEIR [RR'SeL] COMPLEXES

S. N.	Ligand and Compound	N1s		O 1s	S 2p	Se 3p _{1/2}
		Ligand or Uncoord.	Coord.			
1	Ligand L ₁	400.0	–	531.8	–	–
2	[(CH ₃) ₂ Se·L ₁]	–	401.4	531.8	–	167.2
3	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₁]	–	401.4	531.8	–	167.4
4	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₁]	–	401.4	531.8	–	167.4
5	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₁]	–	401.4	531.8	–	167.2
6	[(C ₆ H ₅) ₂ Se·L ₁]	–	401.4	531.8	–	167.2
7	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₁]	–	401.4	531.8	–	167.2
8	[(C ₆ H ₅)(CH ₃)Se·L ₁]	–	401.4	531.8	–	167.4
9	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₁]	–	401.4	531.8	–	167.6
10	Ligand L ₂	400.0	–	531.6	166.2	–
11	[(CH ₃) ₂ Se·L ₂]	–	401.4	531.6	166.2	167.2
12	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₂]	–	401.4	531.6	166.2	167.4
13	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₂]	–	401.4	531.6	166.2	167.4
14	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₂]	–	401.4	531.6	166.2	167.2
15	[(C ₆ H ₅) ₂ Se·L ₂]	–	401.4	531.6	166.2	167.2
16	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₂]	–	401.4	531.6	166.2	167.2
17	[(C ₆ H ₅)(CH ₃)Se·L ₂]	–	401.4	531.6	166.2	167.4
18	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₂]	–	401.4	531.6	166.2	167.6
19	Ligand L ₃	400.0	–	530.6	–	–
20	[(CH ₃) ₂ Se·L ₃]	400.0	–	532.0	–	167.0
21	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₃]	400.0	–	532.0	–	167.2
22	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₃]	400.0	–	532.0	–	167.2
23	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₃]	400.0	–	532.0	–	167.0
24	[(C ₆ H ₅) ₂ Se·L ₃]	400.0	–	532.0	–	167.0
25	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₃]	400.0	–	532.0	–	167.0
26	[(C ₆ H ₅)(CH ₃)Se·L ₃]	400.0	–	532.0	–	167.2
27	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₃]	400.0	–	532.0	–	167.4
28	Ligand L ₄	400.0	–	530.6	–	–
29	[(CH ₃) ₂ Se·L ₄]	400.0	–	532.0	–	167.0
30	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₄]	400.0	–	532.0	–	167.2
31	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₄]	400.0	–	532.0	–	167.2
32	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₄]	400.0	–	532.0	–	167.0
33	[(C ₆ H ₅) ₂ Se·L ₄]	400.0	–	532.0	–	167.0
34	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₄]	400.0	–	532.0	–	167.0
35	[(C ₆ H ₅)(CH ₃)Se·L ₄]	400.0	–	532.0	–	167.2
36	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₄]	400.0	–	532.0	–	167.4
37	Ligand L ₅	400.0	–	530.6	–	–
38	[(CH ₃) ₂ Se·L ₅]	400.0	–	532.0	–	167.0
39	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₅]	400.0	–	532.0	–	167.2
40	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₅]	400.0	–	532.0	–	167.2
41	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₅]	400.0	–	532.0	–	167.0
42	[(C ₆ H ₅) ₂ Se·L ₅]	400.0	–	532.0	–	167.0
43	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₅]	400.0	–	532.0	–	167.0
44	[(C ₆ H ₅)(CH ₃)Se·L ₅]	400.0	–	532.0	–	167.2
45	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₅]	400.0	–	532.0	–	167.4
46	Ligand L ₆	400.0	–	530.6	–	–
47	[(CH ₃) ₂ Se·L ₆]	400.0	–	532.0	–	167.0
48	[(<i>n</i> -C ₄ H ₉) ₂ Se·L ₆]	400.0	–	532.0	–	167.2
49	[(<i>n</i> -C ₈ H ₁₇) ₂ Se·L ₆]	400.0	–	532.0	–	167.2
50	[(<i>i</i> -C ₃ H ₇) ₂ Se·L ₆]	400.0	–	532.0	–	167.0
51	[(C ₆ H ₅) ₂ Se·L ₆]	400.0	–	532.0	–	167.0
52	[(C ₆ H ₅)(C ₂ H ₅)Se·L ₆]	400.0	–	532.0	–	167.0
53	[(C ₆ H ₅)(CH ₃)Se·L ₆]	400.0	–	532.0	–	167.2
54	[(C ₆ H ₅)(CH ₂ Cl)Se·L ₆]	400.0	–	532.0	–	167.4
55	[(CH ₃) ₂ Se]	–	–	–	–	168.8
56	[(<i>n</i> -C ₄ H ₉) ₂ Se]	–	–	–	–	169.0
57	[(<i>n</i> -C ₈ H ₁₇) ₂ Se]	–	–	–	–	169.0
58	[(<i>i</i> -C ₃ H ₇) ₂ Se]	–	–	–	–	168.8
59	[(C ₆ H ₅) ₂ Se]	–	–	–	–	168.8
60	[(C ₆ H ₅)(C ₂ H ₅)Se]	–	–	–	–	168.8
61	[(C ₆ H ₅)(CH ₃)Se]	–	–	–	–	169.0
62	[(C ₆ H ₅)(CH ₂ Cl)Se]	–	–	–	–	169.2

IR spectra of ligand L_3 or L_4 or L_5 or L_6 and their respective [RR'SeL₃₋₆] complexes, $\nu(\text{N-H})$ have not appear in the region 3300-3200 cm^{-1} , suggesting absence of $\nu(\text{N-H})$ group in these [RR'SeL₃₋₆] complexes [34,35]. Furthermore, the bands ~ 230 -260 and ~ 410 -420 cm^{-1} region appeared in all these [(RR'SeL₁₋₁₂)] complexes and are assigned to Se-C and Se-O, respectively [36,37].

The binding energies (eV) data for Se3p_{1/2}, N1s, O1s and S2p_{1/2,3/2} photoelectron peaks are listed in Table-1. The binding energies (eV) for Se3p_{1/2} in RR'Se (where R = R' = CH₃; R = R' = *n*-C₄H₉; R = R' = *i*-C₃H₇; R = R' = *n*-C₈H₁₇; R = R' = C₆H₅; R = C₆H₅, R' = C₂H₅; R = C₆H₅, R' = CH₃ and R = C₆H₅, R' = CH₂Cl) was observed at ~ 168.8 -169.2 eV; while in [RR'SeL] complexes, it was lower (~ 167.0 -167.6 eV) than their respective parent RR'Se complex, which suggest formation of coordinate bond between Se and N or O atom in [RR'SeL] complexes [24] (Figs. 1-3). Furthermore, it was observed that N1s photoelectron binding energy value in L_1 and L_2 were observed at 400 eV; while in their complexes [RR'SeL₁] and [RR'SeL₂] were higher ~ 401.4 eV. Furthermore, it was observed O 1s binding energy values were same in ligand L_1 and [RR'SeL₁] and same way S2p_{1/2,3/2} binding energy value are same as in ligand L_2 and as in [RR'SeL₂] complexes, suggesting non-involvement of oxygen atom in complexation with selenium metal ion in [RR'SeL₁] complexes as well as non-involvement of sulfur atom in complexation with selenium metal ion in [RR'SeL₂] complexes [29] (Table-1).

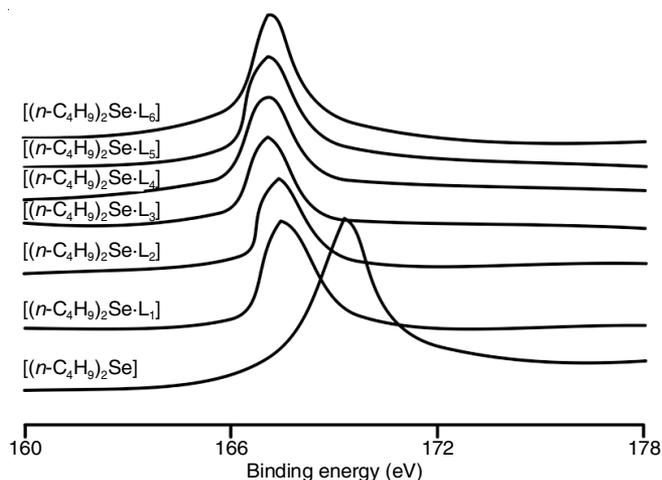


Fig. 1. Se 3p_{1/2} binding energies (eV) in [(*n*-C₄H₉)₂Se·L] and [(*n*-C₄H₉)₂Se·L] complexes

But in case of ligand L_3 or L_4 or L_5 or L_6 ; N1s binding was same in these ligands and [RR'SeL₃₋₆] complexes, suggesting non-involvement of nitrogen atom in complexation with selenium metal ion in these [RR'SeL₃₋₆] complexes (Table-1). Furthermore, it was observed ligand L_3 or L_4 or L_5 or L_6 have shown O1s binding energy at 530.6 eV; while in [RR'SeL₃₋₆] complexes, it was higher *i.e.* 532.0 eV, suggesting oxygen atom with selenium metal ions are involved in coordination in these [RR'SeL₃₋₆] complexes [29] (Table-1).

On the basis of elemental analysis, molar conductivity, IR and XPS data the possible structure of all these [RR'SeL₁₋₆] complexes have been established as an octahedral geometry.

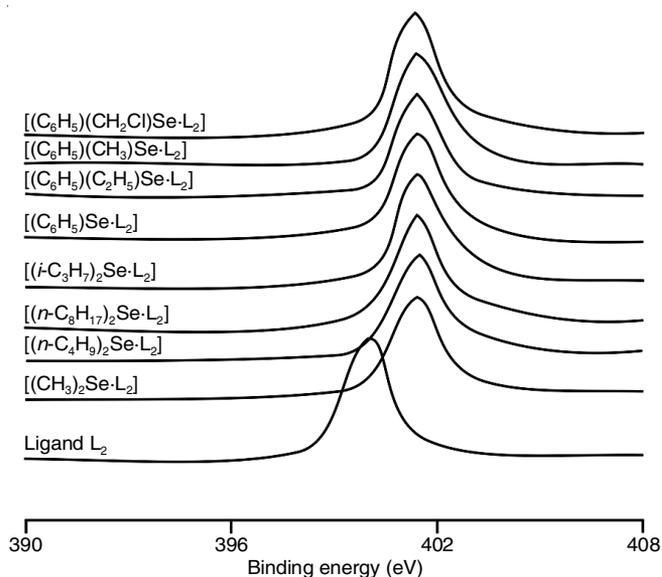


Fig. 2. N1s binding energies (eV) in ligand L_2 and [(RR')Se·L₂] complexes

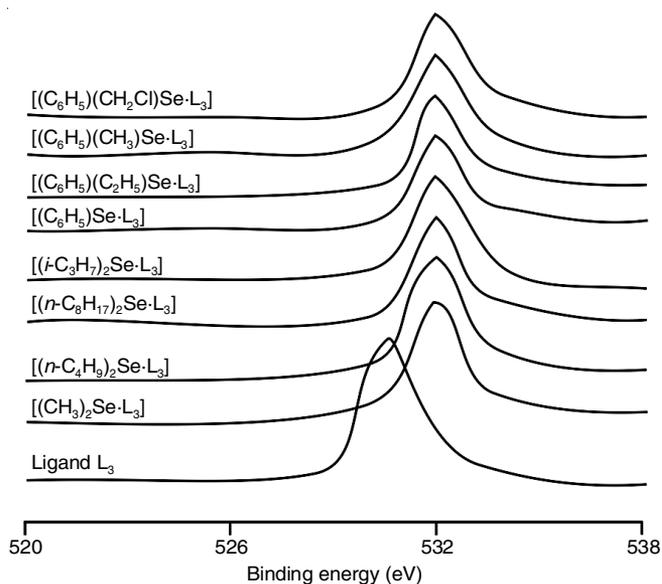


Fig. 3. O 1s binding energies (eV) in ligand L_3 and [(RR')Se·L₃] complexes

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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