**Interesting Selenium-Oxygen/Phosphorus Containing Mixed Donor Macrocycles; Synthetic and Structural Perspectives**

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**Abstract:**

Oxygen, Sulfer-based macrocycles were known and investigated widely. Emergence of the equivalent macrocyclic polyselena ethers was anticipated, since the introduction of sulphur coronands and their extensive and fascinating coordination chemistry. The synthetic along with coordination chemistry investigations of selena-macrocycles have attracted a lot of interest following accidental finding of selena-crown ethers. A lot of work has been put into designing and synthesizing new homo as well as mixed donor selenoether macrocyclic molecules over past few decades in order to examine their ligation characteristics towards transition along with post-transition metal ions. To be able for interaction with both "hard" as well as "soft" guest ions and/or molecules, macrocycles containing soft Se atoms along with other hard donor atoms as binding sites, close to one another within same ring framework, were produced. In this chapter, chemistry of mixed donor (O/P) selena macrocycles has been discussed from their synthetic and structural perspectives, briefly.

**Introduction**:

Since Pedersen1, Cram2, and Lehn's3 pioneering discovery, the science related to "supramolecular chemistry"/"host-guest chemistry" has expanded significantly. The chemistry of associates having an extensively defined framework or chemistry that goes beyond our traditional covalent bonds is both examples of supramolecular chemistry. A number of comparatively weak forces, including H-bonding,  stacking, ion-dipole, dipole-dipole forces, van der Waals interactions, etc., control the architecture of the associated. Chemical sensing, the specific extraction of toxic or radioactive metal ions from streams of waste, membrane transport, the immobilization of radioisotopes, phase-transfer catalysis, and other uses are only a few of the numerous uses involving selective association with metal ions. Prototype platforms for (metallo)enzymes,  activation of small molecules, the specific interaction of anions, as well as the nucleophilic parts of neutral compounds are additional fields of importance. Owing of their extremely specific ability for chelating metal ions, mixed donor macrocycles received a great deal of attention. Bigger Se atom's inclusion ought to alter ring's cavity's dimensions, allowing for certain intriguing coordinating behavior. Additionally, Se-containing macrocycles prove good ligands for binding transition metals and this subject have been thoroughly investigated because of the low electronegativity of Se. Therefore, macrocycles with soft Se atoms and hard donor atoms like Phosphorus, Nitrogen, or Oxygen in the ring structure may function as possible hetero dinuclear macrocyclic ligands. Such macrocyclic ligands are going to possess both 'hard' as well as 'soft' binding sites adjacent to one another, which gives them the ability to communicate with both 'hard' and 'soft' guest ions and/or molecules. The term "hard" describes Group I (alkali metal) as well as Group II (alkaline earth metal) cations that possess poor polarizability. Since these are extremely easy to polarize, transition metal ions are referred to as "soft" cations. The redox characteristics exhibited by the transition-metal ions are going to differ when a hard cation and a transition-metal cation form compounds together within a single macrocycle. These compounds can be utilized for supramolecular network development, activation, along with bimetallic catalysis4. In this chapter selena-macrocyclic systems which additionally have donor centers (Oxygen/Phosphorus) are discussed briefly; from their synthetic, metal complexation and structural viewpoints.

**Selenium-Oxygen donor atom containing macrocycles**:

Se-Se bond-containing organoselenium molecules tend to be interesting as they make good substrates during selected synthetic organic chemistry. These also act as intermediates in crucial biological reactions which involve glutathione peroxidase, which is enzyme that contain Se. when suitable metal cations exist within the cavity, incorporating this bond into crown ether kind hosts offer an outstanding chance for a novel molecular device that can experience specific membrane transport of particular metal cations upon exposure to light or function as a catalyst during a photochemically induced redox process. The *inter-* as well as *intra-*molecular cyclization associated with substrates having Se has been utilised to describe the synthesis of the initial macrocycles with Se-Se linkage (L1, L2), shown in Scheme 1. The noteworthy closeness of Se and O atoms within the structural arrangements of L1 and L2 is an especially intriguing aspect of the structures. The atoms in question have significant attractive interactions with one another, given such seemingly low interatomic spacing. O(12')-Se(1)-Se(2)-O(51) in L1 and Se(6)-Se(5)-O(16) in L2 are in a nearly straight arrangement, which [Figure 1] convincingly demonstrate that those interactions are caused by the hypervalent feature of the Se atoms. The benzene ring and carbonyl group's -electron conjugation as well as the integrated impact of hypervalent interactions all help to maintain the macrocyclic system's structural integrity5.

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| C:\Users\PARTHA\Desktop\Scheme 1.gif | |
| Scheme 1 | |
| C:\Users\PARTHA\Desktop\L1.jpgL1 | C:\Users\PARTHA\Desktop\L2.jpgL2 |
| Figure 1 | |

Three distinct Se4 crown ethers (L3, L4 and L5; n= 1, 2 and 3, respectively) were synthesized using dipotassium benzene-1,2-diselenolate [*o*-C6H4(SeK2)] with dihalogeno selenoethers *o*-C6H4[SeCH2(CH2OCH2)nCH2X]2 (X = Cl, n = 1, 2, 3 or X= Br, n = 3, 4). The yield might be raised by utilizing the "Cs effect" [Scheme 2]6,7. X-Ray diffraction was used to evaluate the crystal framework and solid-state configuration of L4 (Figure 2). The formation of unsymmetrical crown ether modified on just a single benzene ring was additionally done using the "cesium effect."

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| C:\Users\PARTHA\Desktop\Scheme 2_1.png |
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| Scheme 2 |

Two mole equivalents of Mercuric Iodide are bound with these Se-crown ethers to generate dinuclear compounds6 with basic composition as [(HgI2)2(Ln)]; n = 3, 4 and 5. Just those Se donors within the macrocycle connect around the roughly tetrahedral HgII centres, according to the arrangement of [(HgI2)2(Ln)] [Figure 2], while I- donors filling the other two positions for coordination for both HgII center. The synthesis is displayed in Scheme 3, below.

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| C:\Users\PARTHA\Desktop\Scheme 3.pngScheme 3 | |
| C:\Users\PARTHA\Desktop\L4.jpg | C:\Users\PARTHA\Desktop\[(HgI2)2(L5)].jpg |
| L4 | [(HgI2)2(L5)] |
| Figure 2 | |

The photophysics along with electrochemistry properties of the compound [Cu(PPh3)2(L7)](BF4) [Figure 3] were described. The ligand L7 was synthesised as shown in Scheme 4. Electronic absorption as well as emission spectroscopy is used to examine how soft metal ions are encapsulated by the complex's ring. In contrast to the analogous all-oxygen counterpart crown ether complex, that favours binding Gr I and Gr II cations including Na+, K+, and Ba2+, [Cu(PPh3)2(L7)](BF4) favors to interact with transition metal cations including Zn2+ and Cd2+. The inclusion of the softer Se donor element in L7, provides greater affinity towards softer metal cations, is responsible for the changes that have been noted in ion interaction properties8.

Researchers produced L8 using a method9 identical to that used for generating L3-5. The interaction between L8 and Li2[PdCl4] produced the neutral molecule [PdCl2(L8)], where  PdII is most likely linked to a Se2Cl2 combination (Scheme 5). Four Se donors comprising from two Se-macrocyclic molecules are arranged in a square planar pattern surrounding the cation in the framework of the unique Se-cationic PdII compound [Pd(L8)2]X2, [X=NO3, PF6]10. Figure 3, displays the crystal structure of the cation of [Pd(L8)2](PF6)2.

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| C:\Users\PARTHA\Desktop\Scheme 4.gif | |
| Scheme 4 | |
| Cation of [Cu(PPh3)2(L7)](BF4)C:\Users\PARTHA\Desktop\[Cu(PPh3)2(L7)](BF4).jpg | C:\Users\PARTHA\Desktop\[Pd(L9)2](PF6)2.jpgCation of [Pd(L8)2](PF6)2 |
| Figure 3 | |
| E:\Partha Pratim Das_02-09-2023\Book Chapter\New Projects\S,Se, Te macrocycles\Mixed Donor_Se_O_N_P\Scheme 5.pngScheme 5 | |

The cation binding properties of a number of Se4-crown ether analogues using aqueous Group I (Li+, Na+, K+) as well as certain heavy (Ag+, Tl+) metal cations are assessed11,12,13. The ligands involved are displayed in Figure 4. Synthesis method of L10 is shown in Scheme 6.

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| Figure 4 |
| C:\Users\PARTHA\Desktop\Scheme 6.gif |
| Scheme 6 |

By carrying out the study in C2H5OH rather than liquid NH314, it is possible to improve the production of the novel macrocycle L13 [Scheme 7]. The reaction involving 1,2-bis(2-bromoethoxy)ethane, 1,2-diselenocyanatoethane, and sodium borohydride yields the nine-membered ring molecule L14 as a side effect, that was additionally noted as an unanticipated ring contracting result15 [Scheme 7]. As shown in Figure 5, the compound [PtCl2(L13)] exhibits a square planar architecture (Se2Cl2) therefore the PtII core is not interacting with any ether oxygen donors.

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| Scheme 7 | |
| C:\Users\PARTHA\Desktop\Figure 5.gif | E:\Partha Pratim Das_27-08-2023\Book Chapter\New Projects\S,Se, Te macrocycles\Mixed Donor_Se_O_N_P\[PtCl2(L14)].jpg |
| Figure 5 | |

According to Scheme 8, two calix[4]crowns with Se functionality, L15 and L16, were produced. In the process of producing L16, a byproduct known as double bridged biscalix[4](tetraseleno)crownether (L17) is produced in 4% production; nevertheless, this amount of product is capable of being boosted by gradually adding the disodium salt of 1,3-propanediselenol into the solution of the corresponding precursor. Figure 6 shows the cone structure that Calix[4]crown ether (L15) exhibits16.

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| E:\Partha Pratim Das_02-09-2023\Book Chapter\New Projects\S,Se, Te macrocycles\Mixed Donor_Se_O_N_P\Scheme 8.pngScheme 8 | |
| C:\Users\PARTHA\Desktop\L16.jpg | C:\Users\PARTHA\Desktop\16-70_p-p stacking.jpg |
| L15 | Self-assembly of L15via parallel-displaced – stacking |
| C:\Users\PARTHA\Desktop\Infinite sheet aggregate of calix[4]crown ether.jpg | |
| Infinite sheet aggregate of calix[4]crown ether L15 | |
| Figure 6 | |

Through the self-inclusion of the two cavities, it generates a dimer-like architecture. Surprisingly, in the solid phase, *inter-*molecular Se-Se interactions allowed this dimeric arrangement to gradually organise forming a 2-D aggregation16 [Figure 6]. These ionophores work effectively for AgI-selective electrodes. Exceptional potentiometric selectivity coefficients for AgI can be obtained by polymer membranes comprising the ionophores L15 and L16. This happens as a result of their strong linkage to Se donor atoms through soft-soft interactions with high hydration energies as opposed to transition metal cations having +2 charges for their fragile interactions with Se donors of the ionophore.

Step-by-step synthesis was used to generate two novel Se-crown ethers, L18 and L19 [Scheme 9]. UV-Vis spectroscopy was used for calculating the complex stability constants as well as thermodynamic variables related to the interaction of C60 with various crown ethers, resulting L19 having the highest Ks value. The association of C60 to L18 and L19 [Scheme 10] is completely enthalpy-driven thermodynamically, whereas the stability of the combination is controlled by the entropy component17.

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| Scheme 10 |

**Selenium-Phosphorus donor atom containing macrocycles:**

The one-pot procedure depicted in Scheme 11 was used to generate the 10 to 17-member macrocycles L21–32, each of which contained one P and two Se donors. A byproduct L20, is additionally separated in each instance18. Furthermore, as shown in Scheme 12, two similar macrocycles L33 and L34 that each contained two ether-O donors within the cycle were published. The phosphoryl P=O bond for all three of the structurally characterised macrocycles (L23, L26, and L34) points upward towards the interior of the macrocycles18. Four sets of two separate molecules, each of which can't be converted into other through a translational as well as symmetrical operation, comprise L23's unit cell. Apart from the dihedral angles among the benzene ring's planes, there are no appreciable variations in the bonding properties between the two molecules. Aside L33, that generates a 2:1 compound, all macrocyles interact with PdCl2 to form 1:1 compounds18, as depicted in Scheme 12. The crystal structures of L23, L26, and L34 are shown in Figure 7. The reported synthesis of metal complexes with these ligands is shown in Scheme 13. Selenium-containing phosphocane L35, forms 2:1 PdII Complex [Pd(L35)2Cl2] with PdCl2. The synthetic method is shown in Scheme 13 and the crystal structure is shown in Figure 7.

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| E:\Partha Pratim Das_02-09-2023\Book Chapter\New Projects\S,Se, Te macrocycles\Mixed Donor_Se_O_N_P\Scheme 22_1.png | | |
| E:\Partha Pratim Das_02-09-2023\Book Chapter\New Projects\S,Se, Te macrocycles\Mixed Donor_Se_O_N_P\Scheme 22_2.pngScheme 12 | | |
| C:\Users\PARTHA\Desktop\Scheme 23_1.gif | | |
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| Scheme 13 | | |
| C:\Users\Hp\Desktop\L40.jpg  L23 | | C:\Users\Hp\Desktop\L43.jpgL26 |
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| L34 | [Pd(L35)2Cl2] | |
| Figure 7 | | |

For [Pd(L35)2Cl2]18, the protons' signals  to the Se atom for L35 (δ = 2.86 and 3.12 ppm) are moved by 0.5-0.6 ppm to the lower field (δ = 3.50 and 3.60 ppm). The drop in electron density over the Se atom following complex formation was the cause for this down-field movement. It must be emphasised that only minor downfield movements (0-0.2 ppm) were seen for the protons of [Pd(L35)2Cl2]  to the O donors, demonstrating PdII binding does not take place on O donors.

The head-to-tail cyclization of the intermediate anion derived from the reaction between the dimer [(Cl)(Se=)P(-NtBu)]2 and Na metal, resulting in Se-bridged hexamer [(Se=)P(-NtBu)2P(-Se)]6 macrocycle19. In Scheme 14, the interaction is depicted. The intermediary anion's ambidentate character is what causes the P-Se-P link to develop. The structural analysis of [(Cl)(Se=)P(-NtBu)]2 and the 31P NMR spectrum studies indicate the existence of *cis-* and *trans-* isomers in a 7:1 ratio. A macrocyclic product is influenced by the amount of the *cis-* isomer of [(Cl)(Se=)P(-NtBu)]2 present. Six [(Se=)P(-NtBu)2P(-Se)] components connected via bridging Se atom, collectively generates a macrocyclic configuration of [(Se=)P(-NtBu)2P(-Se)]6. Just a minor distortion from overall planarity exists in the molecule. The P2N2 ring components give the molecules a toroidal form because they are nearly perpendicular towards the median plane of the macrocycle. A brand-new class of Se-macrocycle; [(Se=)P(-NtBu)2P(-Se)]6, stands out due to its rigid, crown-like structure and enormous cavity diameter. In [(Se=)P(-NtBu)2P(-Se)]6, the -Se donors have a cisoid shape in relation to the P2N2 rings, which makes Se donors accessible for possible metal linkage19. Additionally, *exo-* Se donors have the potential to coordinate to metal ions. Such Se-macrocycle's rigidity as well as sizable molecular cavity render the host-guest interaction especially intriguing. The crystal structure of [(Se=)P(-NtBu)2P(-Se)]6 is shown in Figure 8.

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| C:\Users\PARTHA\Desktop\Scheme 24.png |
| Scheme 14 |
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| Figure 8 |

Massive ring diselenides with the P-Se-Se-P connectivity were reportedly synthesised, as shown in Scheme 15. The eight, nine, and ten-member rings are present, according to the frameworks of L36-38. Each of the molecules contain both R, R as well as S, S enantiomers; no R, S/S, R diastereomers were found. Two phenyl rings are located on the reverse side of the heterocycle, and the macrocyclic structure is strongly puckered. Tetrahedral geometry around both P atoms is deformed20. In Figure 9, their crystal structures are displayed. L39 and L40, two further P-Se heterocyclic diselenides, was additionally documented20. Despite the P-Se-Se angles for L39 as well as L40 are greater compared to that observed in L36-38, the architecture of L39 and L40 resemble the structures of L36-38. In Figure 9, their structures are depicted. Synthesis of L36-38 are also displayed in Scheme 15.

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| C:\Users\PARTHA\Desktop\Scheme 25.gif | |
| Scheme 15 | |
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| L36 | L37 |
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| L38 | L39 |
| C:\Users\PARTHA\Desktop\L57.jpg | |
| L40 | |
| Figure 9 | |

**Conclusion**:

In summary, this chapter addresses chemistry of some important Selenium-Oxygen/ Phosphorus containing macrocycles. Organoselenium compounds with Se-Se (Diselenium) bonds are usually noteworthy because they function diversely as substrates in some synthetic organic chemistry. In important biological processes involving glutathione peroxidase, a selenium-containing enzyme, diselenium components function as intermediates. Keeping that mind, macrocycles with diselenium bonds5 were discussed. Macrocycles with multiple Se, O donors with different ring sizes and their metal complex synthesis have been thoroughly discussed. Two calix[4]crowns with Se functionality and double bridged biscalix[4](tetraseleno)crownether were mentioned. Regarding AgI-selective electrodes, such ionophores function satisfactorily. This occurs as a consequence of their robust association with Se donors via soft-soft connections16. Interaction of C60 with Se-O-crown ethers is intriguing17. Phosphorus containing Se-macrocycles are not common. P/Se and P/O/Se-containing macromolecule’s synthesis, metal complexation were focused18. Diselenides with the P-Se-Se-P connectivity20 and a very unique and rare P/N/Se containing macrocyclic molecule19 [(Se=)P(-NtBu)2P(-Se)]6 are known and their synthesis procedures and structural details are depicted.

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