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Synthetic and Structural Studies of Lithium Complexes of Selenophosphorus Ligands

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The synthesis and characterization of three lithium complexes of selenophosphorus anions are presented: namely, a lithium selenophosphinite [Ph_2PSeLi·TMEDA]$_2$, **1**; a lithium diselenophosphinate [Ph_2PSe_2Li·THF·TMEDA], **2**; and a dilithiated triselenophosphonate complex [(c-C_6H_{11})PSe_3Li_2·2TMEDA], **3**. The selenophosphinite **1** was prepared using the insertion reaction of elemental Se into the P–Li bond of lithiated diphenylphosphine, and this was then oxidized by additional Se to give **2**. Dilithiation of a primary phosphine followed by reaction with 3 equiv of elemental Se gave the triselenophosphonate **3**. X-ray crystallography reveals **1** to be a dimer in the solid state with two different bridging coordination modes exhibited by the selenophosphinite ligands, and **2** and **3** to both be monomeric. In addition, solution studies using multinuclear (^{31}P and ^{77}Se) and variable-temperature NMR show the anionic charge to be predominately localized on the Se in **1**, and fully delocalized over the PSe_2 and PSe_3 moieties respectively in **2** and **3**.

Introduction

Although the chemistry of alkali-metal complexes containing alkoxide and thiolate ligands has been well studied,[1] the chemistry of the heavier group 16 homologues, the selenolates and tellurolates, is still a relatively underdeveloped area and has only recently begun to attract more attention. This recent interest has been prompted by the desire for new and efficient syntheses for metal chalcogenolates for use as precursors to semiconducting selenides and tellurides[2] and also by the search for new reagents for the synthesis of selenium- and tellurium-containing organics.[3]

We have been interested in the s-block metal coordination chemistry of alkyl and aryl selenolates and tellurolates;[4] however, further work in this area has been hampered by the high air sensitivity and pungent nature of these compounds. This has led us to turn our attention to seleno- and tellurophosphorus ligands (specifically chalcogen derivatives of lithiated primary and secondary phosphines) as these species tend not to suffer so much from the instability, toxicity, and foul smell that are often associated with the organo derivatives of Se and Te. In addition, the related thiophosphorus ligands display a wide range of coordination patterns resulting in a great diversity of molecular and supramolecular structures and have received considerable attention in the literature.[5,6] Despite this, only a handful of metal complexes with seleno- or tellurophosphorus ligands have been reported, and to date the only X-ray diffraction study of an alkali-metal complex of such a ligand is that of [$Na_2(Se_2PPh_2)$·THF·$5H_2O$] formed in small yield as one of a mixture of products from the reaction of Ph_2PCl with Na_xSe_y.[7] Alkali-metal complexes of mixed chalcogen dichalcogenophosphinates have also been studied, notably chalcogenoselenophosphinates $R_2P(X)Se^-$ (X = O, S, Se) by Kuchen and Hertel,[8] and chalcogenotellurophosphinates $R_2P(X)Te^-$ (X = O, S, Se, Te) by Bildstein and Sladky.[9]

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10.1021/ic010835q CCC: $22.00

Table 1. Summary of Crystal Structure Data for **1**, **2**, and **3**[a]

	1	**2**	**3**
empirical formula	$C_{36}H_{52}Li_2N_4P_2Se_2$	$C_{22}H_{34}LiN_2OPSe_2$	$C_{39.5}H_{90}Li_4N_8P_2Se_6$
fw	774.56	538.34	1240.66
cryst size (mm)	0.32 × 0.30 × 0.28	0.13 × 0.13 × 0.10	0.15 × 0.12 × 0.12
cryst syst	triclinic	monoclinic	tetragonal
space group	$P\bar{1}$	$P2_1/c$	$I4_1/a$
a (Å)	12.0608(4)	13.9080(8)	37.2074(5)
b (Å)	13.8132(5)	12.3260(9)	
c (Å)	13.8828(4)	15.7120(6)	17.2586(3)
α (deg)	91.934(2)		
β (deg)	113.640(2)	110.988(3)	
γ (deg)	104.794(2)		
V (Å^3)	2024.18(12)	2514.8(3)	23892.6(6)
Z	2	4	16
ρ_{calcd} (g/cm^3)	1.271	1.422	1.380
θ range (deg)	1.62−27.46	2.95−25.05	2.93−25.02
μ (mm^{-1})	1.934	3.019	3.761
no. of reflns	13042	6335	20646
no. of unique reflns (R_{int})	9178 (0.0254)	4364 (0.0360)	10525 (0.0565)
no. of params	453	266	605
R_1 [$F_o^2 > 2\sigma(F_o^2)$][b]	0.0380	0.0372	0.0460
wR_2 (all data)[c]	0.1171	0.0914	0.1057
min/max electron residue (e/Å^3)	+0.818/−0.394	+0.586/−0.510	+0.603/−0.466

[a] Parameters in common: Mo Kα radiation, $\lambda = 0.71073$ Å; temperature = 180(2) K. [b] $R_1 = \sum|F_o| - |F_c|/\sum|F_o|$. [c] $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum(F_o^2)^2]^{1/2}$.

We report here the first crystallographic study of solvated lithium salts of a selenophosphinite anion **1**, a diselenophosphinate anion **2,** and a triselenophosphonate dianion **3**, as well as full structural characterization of these species in solution using multinuclear and variable-temperature NMR spectroscopic studies. In addition, we present a new and high-yielding route to lithium triselenophosphonates such as **3**.

Experimental Section

General Procedures. All reactions and manipulations were carried out under an inert atmosphere of dry nitrogen or argon using standard double manifold and glovebox techniques. Purification and drying of the solvents were done using standard methods.[10] Ph_2PH and $CyPH_2$ were purchased from Strem and all other chemicals from Lancaster.

NMR spectra were recorded on either a Bruker DPX400 (^{1}H and ^{31}P) or a Joel EX270 Delta Upgrade (low-temperature ^{31}P and ^{77}Se) spectrometer. The standards used were TMS (^{1}H, ^{13}C), 85% H_3PO_4 (^{31}P), or Me_2Se (^{77}Se) all referenced to $\delta = 0.0$ ppm. The melting points were measured in sealed capillaries under argon.

Synthesis of [$Ph_2PSeLi\cdot TMEDA$]$_2$, 1. *n*-BuLi (1.6M in hexanes, 3.2 mL, 5 mmol) was added to Ph_2PH (0.86 mL, 5 mmol) and TMEDA (0.75 mL, 5 mmol) in 4 mL of toluene/2 mL of THF. The resulting solution was then transferred onto powdered gray Se (0.40 g, 5 mmol) in 4 mL of toluene at −78 °C and allowed to warm slowly to room temperature to give a clear yellow solution. This solution was subsequently reduced in volume to ca. 5 mL and left for 48 h at 5 °C to yield colorless crystals of **1** (1.37 g, 71%). Mp: 139−140 °C. ^{1}H NMR (250 MHz, C_6D_6): $\delta = 1.74$ (s, 4H; TMEDA), 2.02 (s, 12H; TMEDA), 7.02−8.09 (m, 10H; Ph_2P-). ^{13}C NMR (101 MHz, d_6-DMSO): 148.3, 132.5, 128.7, 126.8 (Ph_2P-), 57.3, 45.6 (TMEDA). ^{31}P NMR (162 MHz, C_6D_6): $\delta = 5.1$ (s + d satellites, $^1J_{PSe} = -316$ Hz). ^{77}Se (51.5 MHz, C_6D_6): $\delta = -233$ (d, $^1J_{PSe} = -321$ Hz). Elemental anal.: calcd C 55.82, H 6.77, N 7.23; found C 54.76, H 6.65, N 7.47.

Synthesis of [$Ph_2PSe_2Li\cdot THF\cdot TMEDA$], 2. *n*-BuLi (1.6 M in hexanes, 3.2 mL, 5 mmol) was added to Ph_2PH (0.86 mL, 5 mmol) and TMEDA (0.75 mL, 5 mmol) in 2 mL of toluene/4 mL of THF, then reacted with a suspension of powdered gray Se (0.80 g, 10 mmol) in 4 mL of toluene at −78 °C, and allowed to warm slowly to room temperature to give a clear yellow solution. Crystals of **2** grew from this solution at room temperature over 24 h in 91% yield (2.46 g). Mp: 151−153 °C. ^{1}H NMR (250 MHz, C_6D_6): $\delta = 1.50$ (m, 4H; THF), 1.80 (s, 4H; TMEDA), 2.01 (s, 12H; TMEDA), 3.66 (m, 4H; THF), 7.01−8.69 (m, 10H; Ph_2P-). ^{13}C NMR (101 MHz, d_6-DMSO): 143.5, 130.9, 128.4, 126.7 (Ph_2P-), 67.1 (THF), 57.3, 45.6 (TMEDA), 25.2 (THF). ^{31}P NMR (162 MHz, C_6D_6): $\delta = 20.7$ (s + d satellites, $^1J_{PSe} = -585$ Hz). ^{77}Se (51.5 MHz, C_6D_6): $\delta = -41$ (d, $^1J_{PSe} = -578$ Hz). Elemental anal.: calcd C 49.08, H 6.37, N 5.20, P 5.75; found C 48.84, H 6.40, N 5.24, P 5.73.

Synthesis of [$CyPSe_3Li_2\cdot 2TMEDA$], 3. *n*-BuLi (1.6 M in hexanes, 3.2 mL, 5 mmol) was added to $CyPH_2$ (0.33 mL, 2.5 mmol) and TMEDA (0.75 mL, 5 mmol) in 4 mL of toluene/4 mL of THF. This solution was then reacted with powdered gray Se (0.60 g, 7.5 mmol) in 4 mL of toluene at −78 °C and allowed to warm slowly to room temperature to give a clear yellow solution, which was then reduced in volume to give a yellow oil before addition of 2 mL of hexane and 3 mL of toluene. Crystals of **3** grew from this solution over 7 days at −30 °C in 81% yield (1.26 g). Mp: 98−101 °C. ^{1}H NMR (250 MHz, C_6D_6): $\delta = 0.90-2.46$ (m, 11H; $C_6H_{11}-$), 1.89 (s, 8H; TMEDA), 2.21 (s, 24H; TMEDA), 2.24 (s, 3H; toluene), 7.14 (m, 5H; toluene). ^{13}C NMR (101 MHz, d_6-DMSO): 57.6 (TMEDA), 56.6 ($C_6H_{11}-$), 46.0 (TMEDA), 28.3, 27.8, 27.2, 27.0, 26.4 ($C_6H_{11}-$). ^{31}P (162 MHz, C_6D_6): $\delta = 7.27$ (s + d satellites, $^1J_{PSe} = -477$ Hz). ^{77}Se (51.5 MHz, C_6D_6): $\delta = 166$ (d, $^1J_{PSe} = -474$ Hz). Elemental anal.: calcd C 37.24, H 7.28, N 9.20 for $\mathbf{3}\cdot{}^1/_4$toluene; found C 36.96, H 7.19, N 8.82.

X-ray Structure Determinations of 1, 2, and 3. The crystals were all taken directly from the mother liquor, covered with a perfluorinated ether, and mounted on the top of a glass capillary under the flow of cold gaseous nitrogen. The data for all three crystals were collected on a Nonius Kappa CCD diffractometer equipped with an Oxford Cryostream low-temperature device.

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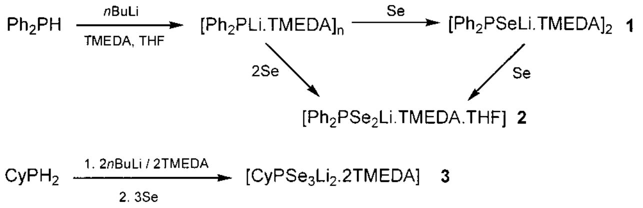
Scheme 1. The Synthesis of Complexes **1**, **2**, and **3**



Crystal data and other information on the structure determination procedures are given in Table 1.

The structures were solved by direct methods,[11] and refinement, based on F^2, was by full-matrix least-squares techniques,[12] with weighting $w^{-1} = [\sigma^2(F_o^2) + (aP)^2 + (bP)]$, where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined with anisotropic displacement parameters, and isotropic H atoms were constrained with a riding model. Disorder was found in the structures of both **1** and **3**. Two alternative positions were resolved and refined for three of the carbon atoms in one of the TMEDA ligands in **1**, with occupancy factors 0.64:0.36(1); five carbons of the cyclohexyl group on one of the two molecules in the asymmetric unit of **3**, 0.80:0.20(1); and three carbons on one of the TMEDA groups of the same molecule in **3**, 0.59:0.41(1). Restraints were applied to geometry and displacement parameters in the disordered groups.

Results and Discussion

Treatment of lithiated diphenylphosphine with 1 equiv of elemental Se in the presence of the Lewis base donor TMEDA (tetramethylethylenediamine) gave Se insertion into the P−Li bond to form $[Ph_2PSeLi{\cdot}TMEDA]_2$, **1** in high yield. This lithium selenophosphinite was then oxidized by addition of another equivalent of Se to give the lithium diselenophosphinate $[Ph_2PSe_2Li{\cdot}TMEDA{\cdot}THF]$, **2**. However, better yields of **2** were achieved from reaction of the initial lithiated diphenylphosphine with 2 equiv of Se (Scheme 1). Such Se insertion reactions into polar R−M bonds have been known for many years,[13] and have proved to be one of the most valuable methods for the synthesis of organic selenolates since they do not require the use of readily oxidized selenols as starting materials.[3] Indeed, reaction of metalated diorganylphosphides with elemental chalcogens to give dichalcogenophosphinates has already been employed in the synthesis of chalcogenotellurophosphinates.[9] Another published route to diselenophosphinates is the reaction of chlorophosphines, R_2PCl, first with selenium to give $R_2P(Se)Cl$ and then with 2 equiv of NaSeH to give the sodium salt of the diselenophosphinate and hydrogen selenide.[8,14]

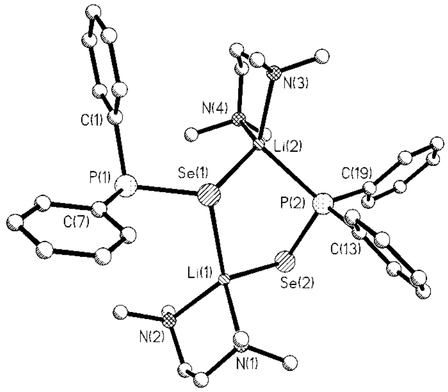


Figure 1. Solid-state structure of $[Ph_2PSeLi{\cdot}TMEDA]_2$, **1**. Hydrogen atoms and disorder in one of the TMEDA ligands are omitted for clarity.

Table 2. Selected Bond Lengths (Å) and Angles (deg) for Complex **1**

P(1)−Se(1)	2.233(1)	P(2)−Se(2)	2.200(8)
Se(1)−Li(1)	2.623(4)	Se(2)−Li(1)	2.589(5)
Se(1)−Li(2)	2.564(5)	P(2)−Li(2)	2.508(5)
P(1)−Se(1)−Li(1)	103.60(11)	P(2)−Se(2)−Li(1)	95.52(11)
P(1)−Se(1)−Li(2)	115.59(12)	Se(2)−P(2)−Li(2)	103.39(12)
Se(1)−Li(1)−Se(2)	108.03(17)	Se(1)−Li(2)−P(2)	97.30(17)
Li(1)−Se(1)−Li(2)	95.66(15)		

We then attempted to extend the synthetic procedure to the reaction of 3 equiv of elemental Se with a dilithiated primary phosphine, $CyPLi_2$ (Cy = c-C_6H_5). As was hoped, two Se inserted into the P−Li bonds and the third Se oxidized the phosphorus(III) to phosphorus(V) to yield the dilithium triselenophosphonate complex $[CyPSe_3Li_2{\cdot}2TMEDA]$, **3** (Scheme 1). It is interesting to note that use of less than 3 equiv of Se also resulted in the synthesis of **3**, albeit in lower yield. Currently the only other characterized examples of triselenophosphinate complexes are the platinum salts synthesized from the reaction of $(PhPSe_2)_2$ with $PtCl_2(PR_3)_2$.[15] However Karaghiosoff has indicated that these species are also accessible from reaction of $RPCl_2$ with Na_2Se and Na_2Se_2 in a 1:1:1 ratio.[16]

All of the complexes **1**−**3** were isolated as colorless air-sensitive crystalline solids. X-ray diffraction studies show **1** to dimerize in the solid state via a five-membered P−Se−Li−Se−Li ring (Figure 1) with each lithium atom bis-coordinated by one TMEDA molecule. Two different coordination modes are displayed by the selenophosphinite ligands in this structure; end-on bridging from Se to two Li and side-on bridging from Se to one Li and P to another Li. The P−Se bond lengths (2.233(1) and 2.200(8) Å; Table 2) within the selenophosphinite units are consistent with P−Se single bonds,[17] with the side-on bridging unit having the slightly shorter bond. This suggests that the negative charge is essentially located on the selenium atoms in $Ph_2P{-}Se^-$ units, with negligible contribution from the $Ph_2P({=}Se)^-$ tautomer. The localization of the negative charge on the Se is also reflected in the high upfield shift ($\delta = -233$ ppm) of the doublet in the ^{77}Se NMR of **1**. This behavior therefore contrasts with that reported for the conjugate acids of chalcogenophosphinites which exist almost exclusively in their secondary phosphine chalcogenide tautomer, $[R_2P(X)H$, X = O, S, Se], and hence contain a P−Se double bond for all but the electron withdrawing CF_3 substituted compounds

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[17] Mean P−Se single bond length of 25 structures from the Cambridge Crystallographic Structural Database, 2.26 Å.

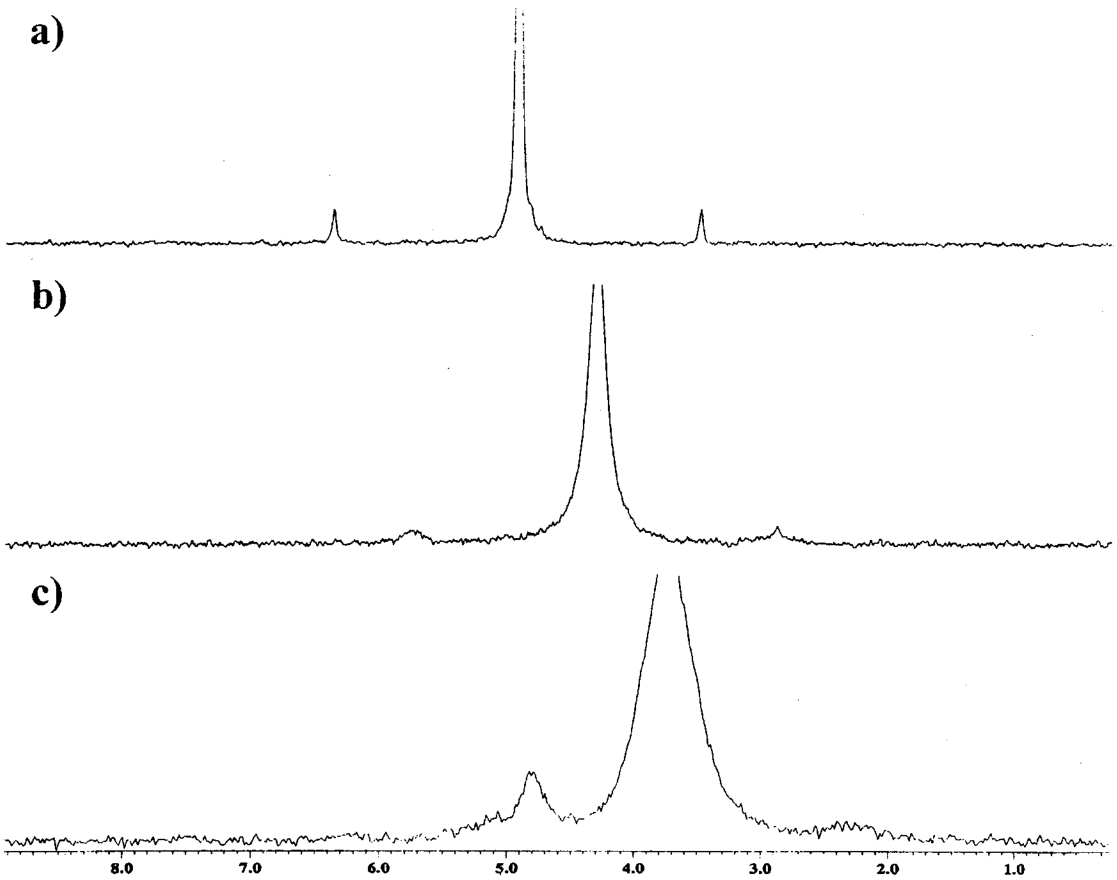

Figure 2. ^{31}P NMR spectra of **1** at (a) 15 °C, (b) −40 °C, and (c) −70 °C. The central ^{31}P peak is truncated for clarity in each case.

which exist as their chalcogenophosphinous tautomers (R_2PXH; R = CF_3; X = O, S).[5]

We attempted to observe the presence of the two coordination modes of the selenophosphinite in solution as well as in the solid state using ^{31}P NMR. At room temperature the ^{31}P NMR spectra of **1** consisted of only one peak with a pair of ^{77}Se satellites ($^1J_{PSe}$ = −314 Hz), but as the temperature of the solution was lowered the satellites became broader, and at −70 °C the main peak was observed to split into two unequal peaks (see Figure 2). This leads us to believe that a dynamic equilibrium is occurring between the two coordination modes at room temperature which can be frozen out at low temperatures. It has been shown that $^1J_{PSe}$ coupling constants can be directly related to P−Se bond length and the coupling constant in this case is well within the range expected for a P−Se single bond (−200 to −620 Hz).[18] Although the ambivalent character of anionic R_2PE^- (E = O, S, Se, Te) ligands has attracted much interest in the past, the vast majority of studies have concentrated on the oxygen and sulfur derivatives,[5] with the only other solid-state structural characterization of a selenium-based homologue being $(CO)_4Mn[SeP(c\text{-}C_6H_{11})_2]$ in which the phosphoselenolate ligand adopts an η^2 coordination mode to the metal center.[19]

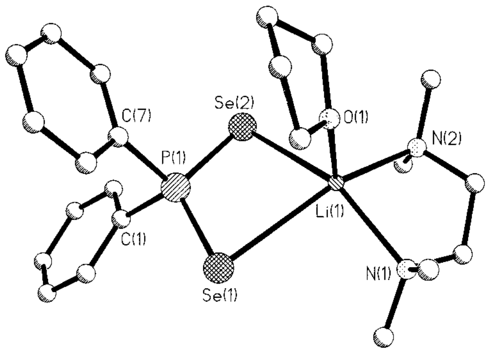

Figure 3. Solid-state structure of [Ph_2PSe_2Li·THF·TMEDA], **2**. Hydrogen atoms are omitted for clarity.

The solid-state structure of **2** is monomeric with the lithium cation coordinated asymmetrically by one THF and one TMEDA molecule (Figure 3). The solid-state structures of three other complexes containing this ligand have previously been reported in the literature: [$Na_2(Se_2PPh_2)$·THF·$5H_2O$],[7] [$Ni(Se_2PPh_2)_2$],[20] and [$Cp_2Ti(Se_2PPh_2)$].[21] In common with these previously reported examples the two P−Se bonds in **2** are approximately the same length [2.141(1) and 2.147(1) Å; Table 3] and are of a value between those expected for a P−Se single[17] and double bond,[22] indicating complete delocalization of the negative charge throughout the PSe_2

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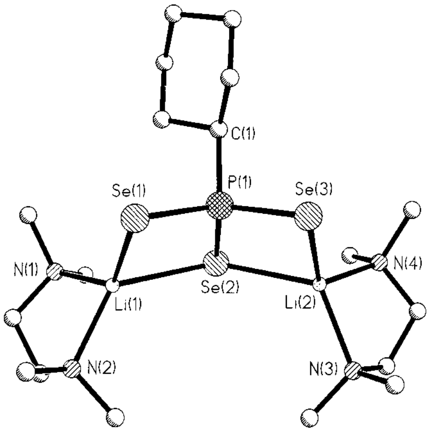

Figure 4. Solid-state structure of [(c-C_6H_{11})PSe_3Li_2·2TMEDA], **3**. Only one of the monomeric molecules in the asymmetric unit is shown. Hydrogen atoms and disorder in the TMEDA ligands are omitted for clarity.

Table 3. Selected Bond Lengths (Å) and Angles (deg) for Complex **2**

P(1)−Se(1)	2.141(1)	P(1)−Se(2)	2.147(1)
Se(1)−Li(1)	2.940(6)	Se(2)−Li(1)	2.848(7)
P(1)−C(1)	1.831(3)	P(1)−C(7)	1.839(3)
Se(1)−P(1)−Se(2)	115.13(4)	Se(1)−Li(1)−Se(2)	77.38(15)
P(1)−Se(1)−Li(1)	80.99(13)	P(1)−Se(2)−Li(1)	83.13(12)
C(1)−P(1)−Se(1)	109.62(11)	C(7)−P(1)−Se(1)	110.43(12)
C(1)−P(1)−Se(2)	109.56(12)	C(7)−P(1)−Se(2)	109.93(10)
C(1)−P(1)−C(7)	101.28(14)		

moiety. The 5-coordinate Li cation in **2** sits asymmetrically between the two Se of the diselenophosphinate [Se(1)−Li(1) 2.940(6) Å, Se(2)−Li(1) 2.848(7) Å]. The discrepancy in these Se−Li bond lengths can be attributed to the steric demands of the two different Lewis base donors about the Li. These Li−Se bond distances are much longer than those observed in **1** and are in fact the longest Li−Se bonds yet observed in the solid state by a considerable amount, the previous longest being 2.7(1) Å in [$Cp_2Lu(\mu\text{-}SeC_6H_5)_2Li(THF)_2$].[22] Their length can be rationalized by a combination the high coordination number (5) of the lithium and the weak bonding of the delocalized PSe_2^- moiety. The ^{31}P NMR spectrum of **2** consists of a single peak with ^{77}Se satellites and a $^1J_{PSe}$ coupling of −585 Hz. The magnitude of this coupling constant is between previously reported common $^1J_{P-Se}$ and $^1J_{P=Se}$ values and therefore tallies with a P−Se bond order of $1^1/_2$.[18] The doublet in the ^{77}Se NMR spectra of **2** is much further downfield (at $\delta = -41$ ppm) than that observed for **1** since the Se atoms are now less well shielded only formally supporting a $^1/_2$ negative charge each.

Crystallographic studies of **3** reveal a tetragonal cell with two structurally identical molecules in the asymmetric unit (one of which is shown in Figure 4). The phosphorus in **3** is in an approximately tetrahedral environment, bonding to three selenium atoms and one carbon. Each Li is coordinated to two Se atoms resulting in one of the Se atoms bridging two Li atoms and the other two Se atoms coordinating terminally to just one Li each. The Se−Li bonds in **3** are all of similar length [mean 2.561 Å, range 2.539(10)−2.585(9) Å; Table 4]. However, two different P−Se bond lengths are observed within each dianionic unit: two short P−Se(terminal) bonds (mean 2.17 Å) and one slightly longer P−Se(bridging) bond (mean 2.21 Å). All the P−Se bonds are therefore longer than expected for P−Se double bonds and also slightly longer than the delocalized P−Se bonds observed in **2**, but shorter than expected for a P−Se single bond such as in **1**. This suggests a bond order of $1^1/_3$ and is indicative of complete delocalization of the dianionic charge over the PSe_3 unit. This therefore contrasts to the only other published example of a triselenophosphonate dianion present in [Pt{$Se_2P(Se)$-PPh}(dppe)]·CH_2Cl_2, in which the phosphoselenolate bonds to the metal center through two Se, each of which is singly bonded to the phosphorus, leaving the third Se doubly bonded to the phosphorus, hence giving one short and two long P−Se distances and no delocalization.[15] The delocalization of the double negative charge in **3** was also observed in solution using ^{31}P NMR spectroscopy since only one set of ^{77}Se satellites were observed even at low temperatures (down to −70 °C). In addition, the $^1J_{PSe}$ coupling constant (−479 Hz) is of a value consistent with a bond order of $1^1/_3$, being between the values reported for **1** and **2**.

Table 4. Selected Bond Lengths (Å) and Angles (deg) for Complex **3**

P(1)−Se(1)	2.177(1)	Se(1)−Li(1)	2.579(8)
P(1)−Se(2)	2.206(1)	Se(2)−Li(1)	2.573(8)
P(1)−Se(3)	2.171(1)	Se(2)−Li(2)	2.549(9)
P(1)−C(1)	1.857(5)	Se(3)−Li(2)	2.539(10)
C(1)−P(1)−Se(1)	108.67(19)	P(1)−Se(1)−Li(1)	80.08(18)
C(1)−P(1)−Se(2)	104.34(18)	P(1)−Se(2)−Li(1)	79.67(18)
C(1)−P(1)−Se(3)	107.19(17)	P(1)−Se(2)−Li(2)	79.5(2)
Se(1)−P(1)−Se(2)	110.85(5)	P(1)−Se(3)−Li(2)	80.3(2)
Se(2)−P(1)−Se(3)	110.26(6)	Se(3)−P(1)−Se(1)	114.92(6)

Concluding Remarks

This work has presented a new and high-yielding route to triselenophosphonate ligand systems in the form of their lithium salts, and has also provided an insight into the coordination chemistry of a series of anionic selenophosphorus ligands. We now hope to use these and related lithium complexes in transmetalation reactions so as to investigate the coordination chemistry of these ligands with other metal centers. We are particularly interested at looking at the use of these ligands in single-source precursors for MOCVD (metal organic chemical vapor deposition) processes toward group 16 containing semiconducting materials.

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Supporting Information Available: Crystallographic information in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

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(22) Mean P−Se double bond length from literature values = 2.093(19) Å. See: *Handbook of Chemistry and Physics*, 75th ed.; Lide, D. R., Ed.; CRC Press: Boca Raton, FL, 1995; pp 9−12.

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