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Synthesis and Structural Characterization of $[Te\{(SePPh_2)_2N\}_2]$ and $[4\text{-}MeOPhTe\{(SPPh_2)_2N\}]_2$

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ABSTRACT: *Reaction of $K[(SePPh_2)_2N]$ with $[Te(tu)_4]Cl_2$ (tu = thiourea) in methanol gave $[Te\{(SePPh_2)_2N\}_2]$, (***1***), while reaction of $K[(SPPh_2)_2N]$ with 4-$MeOPhTeCl_3$ in methanol gave $[4\text{-}MeOPhTe\{(SPPh_2)_2N\}]_2$, (***2***). The crystals of* **1** *are triclinic and contain centrosymmetric molecules. The ligands are bidentate and form a true square planar Te(II) complex with $TeSe_4$ coordination. For* **2**, *the crystals are also triclinic, and the dimeric molecules are centrosymmetric. Each dithiolate ligand bridges the two Te(II) atoms; the result is a T-shaped coordination around each tellurium atom. A tendency to weak Te—Te bonding across the ring-formed dimer roughly in the direction of the missing fourth ligand in a potential square planar tellurium coordination sphere is also found [Te—Te = 3.761 (1) Å]. In* **1**, *the Te–Se bonds have an average length of 2.797 Å. The Te–S bonds in* **2** *are quite asymmetric with Te(1)–S(1) = 2.551 (3) Å and Te(1)–S(2a) = 2.873 (3) Å, while Te(1)–C(1) = 2.10 (1) Å.* © 1998 John Wiley & Sons, Inc. Heteroatom Chem 9: 615–621, 1998

INTRODUCTION

The large-bite imidotetraphenyldichalcogenodiphosphinato anions are versatile ligands. A series of complexes of $(EPPh_2)_2N^-$, where E = O, S, Se, are known with both transition metals and main group elements [1–7]. Usually, these ligands are bidentate, forming six-membered ME_2P_2N rings. The large E-E′ bites aid in forming regular coordination spheres where large central atoms are involved [1,3,8]. Similar ligands with alkyl or alkoxy substituents on phosphorus instead of phenyl groups are also known [4,5]. Monoanionic bidentate ligands with small bites like dialkyldithiophosphates, dialkyldithiophosphinates, dialkyldithiocarbamates, and alkylxanthates are also well known from coordination chemistry. However, with large main group elements like Se, Te, Sn, Pb, As, Sb, and Bi, these ligands are usually anisobidentate [9–12]. This has been attributed to the small bite of these ligands [10,11]. One result of this is the trapezoid planar coordination of Te(II) complexes with such dithiolate ligands [9]. The $R_2Sn(IV)$ group is isoelectronic with Te(II), and this group adds two such ligands, forming a trapezoid planar SnS_4 group in addition to the two skewed Sn–C bonds [3]. Likewise, the R–Sb(III) group will form a similar SbS_4 group at right angles to the Sb–C bond [13].

It was postulated that, with larger-bite ligands,

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the trapezoid planar MS_4(M = Se,Te) coordination should change toward a regular square planar one [10,11]. This was verified for Te(II) with L = $(SPPh_2)_2N^-$ [1] and for $R_2Sn(IV)$ with the same ligand [3]. This ligand is similar to acetylacetonate but is more flexible due to the easy ionization of the proton on nitrogen yielding a monoanion with the charge delocalized over the SPNPS skeleton. The recent preparation of the corresponding diseleno ligand and subsequently of its first metal complexes [14,15] gave us a new tool to probe the coordination behavior of tellurium. In addition, we were interested in the reaction between ArTeX, and such large-bite ligands, hoping to find less distorted structures than those found in complexes obtained by reaction with dithiocarbamates and other small-bite dithiolates [16]. For example, the structure of $PhTeL_3$ (L = $Et_2NCS_2^-$) is greatly distorted pentagonal bipyramid. The unique L spanning one equatorial and one axial position (Ph is also axial) is there strongly anisobidentate and thus responsible for the greatest distortion from a regular symmetry [17]. A large-bite dithiolate ligand may span both an equatorial and an axial position without such distortion and clarify the stereochemical role of the lone electron pair on tellurium in such complexes.

RESULTS AND DISCUSSION

Synthesis

The synthesis of **1** was a simple substitution reaction, but the synthesis of **2** was more complex. The intended reaction was 4-$MeOPhTeCl_3$ + KL = 4-$MeOPhTeCl_2L$, where L = dithiolate ligand. Instead, the following reaction probably took place: 2(4-$MeOPhTeCl_3$) + 6KL = [4-MeOPhTeL]$_2$ + 6KCl + $2L_2$. This is a disproportionation reaction where Te(IV) has been reduced to Te(II) and L has been oxidized to the corresponding disulfide, L_2. Such disproportionation is quite common for Te(IV) compounds in reactions with sulfur-containing ligands, except for dialkyldithiocarbamates [9].

Molecular Structure

The molecular structures of **1** and **2** are shown in Figures 1 and 2, respectively. Atomic coordinates are shown in Tables 1 and 2, and selected bond lengths and angles are shown in Tables 3 and 4. From Figure 1, it is seen that **1** has a central square planar $TeSe_4$ group. There are two crystallographically independent half molecules in the asymmetric unit. These molecules are centrosymmetric and very similar. The bidentate ligands form six-membered rings with the central tellurium atoms. These heterocyclic rings have a chair form in molecule **1A** and a boat form in **1B**; however, the nitrogen atoms are nearly coplanar with the P and Se atoms of the rings in both. The main difference between the molecules is found in the Te-Se-P angles.

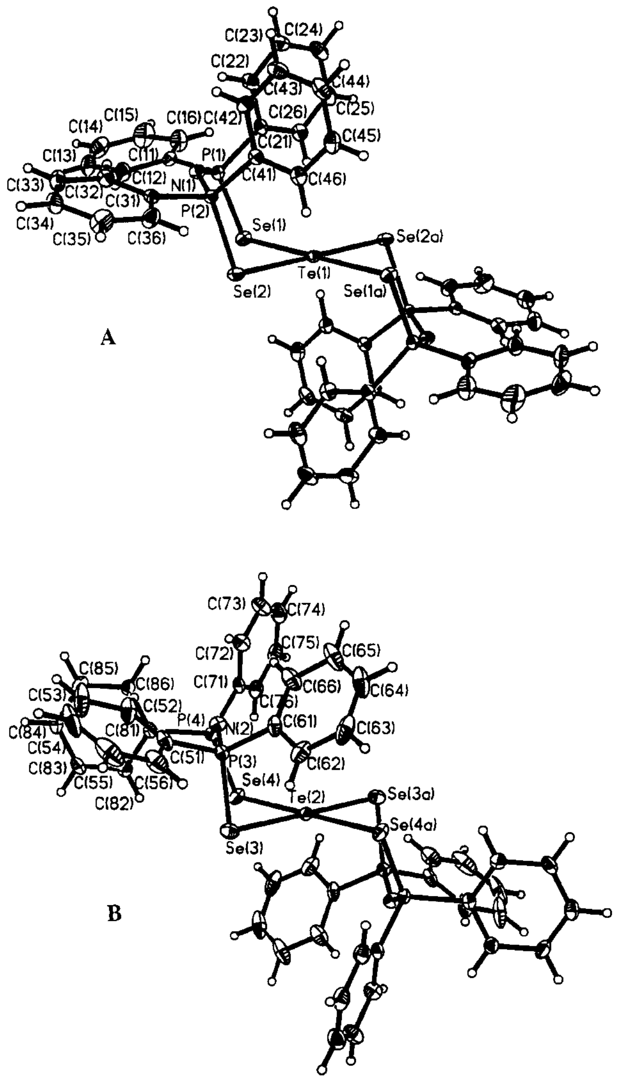

FIGURE 1 The structure of the centrosymmetric molecules A and B of [Te{$(SePPh_2)_2N$}$_2$], **1.** Thermal ellipsoids are given at the 50% probability level. H atoms are represented by spheres of arbitrary radius.

It is not surprising that the crystals of **1** are isomorphous with those of the corresponding thio complex, **3**, which was the first true square planar tellurium complex with a bidentate ligand [1]. The structure of **1** corroborates the idea that large-bite ligands yield more regular structures than ligands with small bites. The molecules of **2** (Figure 2) are dimers and form puckered 12-membered rings. Both dithio ligands bridge two different 4-MeOPhTe groups by forming two greatly asymmetric Te–S

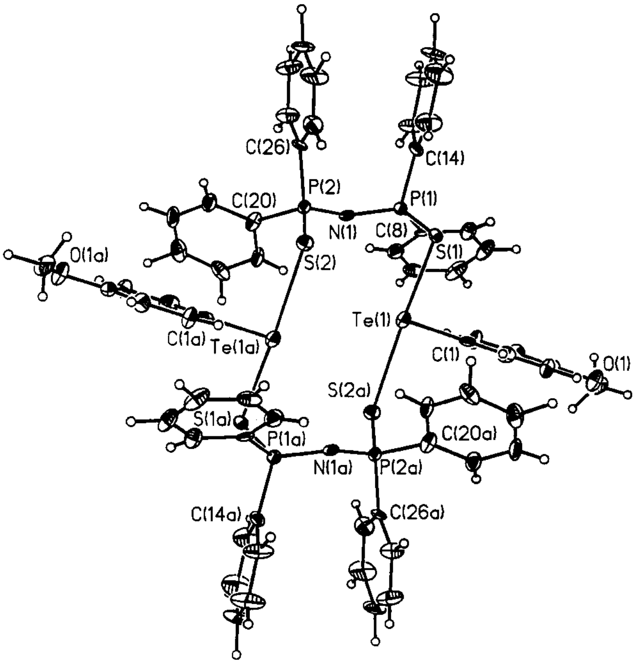

FIGURE 2 The structure of the dimeric, centrosymmetric complex [4-MeOPhTe{(SPPh$_2$)$_2$N}]$_2$, **2.** Thermal ellipsoids are given at the 50% probability level. H atoms are represented by spheres of arbitrary radius.

TABLE 1 Selected Atomic Coordinates and Equivalent Isotropic Temperature Factors for [Te{(SePPh$_2$)$_2$N}$_2$], **1**

Atom	x	y	z	U^*_{eq}
Te(1)	0	0	0	0.01308
Te(2)	0	0.5	0.5	0.01499
Se(1)	−0.12735(5)	0.03001(3)	0.12278(2)	0.01755(11)
Se(2)	0.23797(5)	0.13106(3)	0.07374(2)	0.01731(10)
Se(3)	−0.20999(5)	0.53619(3)	0.39080(3)	0.02260(11)
Se(4)	0.18376(5)	0.58661(3)	0.40276(2)	0.02035(11)
P(1)	−0.12871(12)	0.19366(8)	0.10448(6)	0.0132(2)
P(2)	0.15292(11)	0.27104(8)	0.06596(6)	0.0126(2)
P(3)	−0.15456(12)	0.69877(8)	0.42816(6)	0.0154(2)
P(4)	0.14404(12)	0.74375(8)	0.41083(6)	0.0144(2)
N(1)	0.0145(4)	0.2725(3)	0.0995(2)	0.0159(7)
N(2)	0.0015(4)	0.7581(3)	0.4333(2)	0.0185(8)

U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

TABLE 2 Selected Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **2**·3CH$_2$Cl$_2$

	x	y	z	U(eq)
Te(1)	0.0603(1)	0.1191(1)	0.0311(1)	0.019(1)
P(1)	0.0013(3)	0.1206(2)	0.2722(2)	0.015(1)
P(2)	0.1440(3)	−0.0271(2)	0.2450(2)	0.015(1)
S(1)	0.0654(3)	0.2626(2)	0.1876(2)	0.018(1)
S(2)	0.2269(3)	0.0475(2)	0.1406(2)	0.019(1)
N(1)	0.0163(8)	−0.0054(8)	0.2560(5)	0.016(2)
C(1)	0.1959(11)	0.2001(9)	0.0067(6)	0.021(2)

U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

bonds. The resulting binuclear complex is centrosymmetric, and each tellurium atom has a T-shaped coordination sphere.

Tellurium Ligand Bonding

The Te–Se bonds have slightly different lengths in both molecules of **1**, the short ones being Te(1)–Se(1) = 2.7753 (8) and Te(2)–Se(4) = 2.7895 (10) Å. The long ones are Te(1)–Se(2) = 2.8065 (12) and Te(2)–Se(3) = 2.8153 (10) Å. Similar differences were found for the corresponding Te–S bond lengths in **3.** The average Te–Se bond length in **1** is 2.797 Å. This is 0.26 Å longer than the sum of the covalent radii of Te and Se (2.54 Å) [18]. In linear L-Te-L three-center four-electron bonding (L = chalcogen and halogen ligands), the bond lengthening is on the average 0.27 Å [9] in good agreement with the present investigation. Also square planar Te(II) complexes with monodentate seleno ligands with linear Se-Te-Se three-center, four-electron systems have similar Te–Se bond lengths [19–21]. The only such square planar Te complex with bidentate ligands known previously is [Te(Se$_5$)$_2$]$^{2-}$ [22]. There the average Te–Se bond length is 2.818 (7) Å, while the intraligand Se-Te-Se angle is 102.6 (1)°. The intraligand angles in **1** have an average value of 86.72° compared to 86.80 in the thio analogue (**3**). Here it appears that the greater bite of the seleno ligand relative to that of the thio ligand has no influence on the intraligand S(e)-Te-S(e) angle, but then these ligands are very flexible [1]. The structure of the Sn (II) complex is very similar to that of I [7]. In **2**, the Te(1)–C(1) bond makes a near 90° angle with the nearly linear S(1)-Te(1)-S(2a) [174.20 (8)°] system. The bond lengths are Te(1)–C(1) = 2.100 (1), Te(1)–S(1) = 2.551 (3), and Te(1)–S(2a) = 2.873 (3) Å. We may include the short intramolecular Te(1) Te(1a) contact across the ring {Te(1)–Te(1a) = 3.761 (1) Å compared to a van der Waals contact of 4.12 Å [23]} in the coordination sphere. The result is a distorted square planar coordination sphere, a structure often found with T-shaped tellurium(II) complexes with an aromatic ligand. It is typical that the weak contact is *trans* to the aryl group that has a very strong *trans* influence [9]. Another and weaker contact across the ring is Te(1)—S(2). Its length is 3.733 (3) Å, 0.13 Å less than the corresponding van der Waal's contact [23], and it points in the direction of a fifth position in a distorted pentagonal planar coordination sphere. Tendency to planar five coordination has been found

TABLE 3 Selected Bond Lengths/Å and Angles/° for **1**

Molecule A		*Molecule*	
Te(1)–Se(1)	2.7754(8)	Te(2)–Se(3)	2.7895(10)
Te(1)–Se(2)	2.8065(12)	Te(2)–Se(4)	2.8153(10)
Se(1)–P(1)	2.1756(12)	Se(3)–P(3)	2.1716(13)
Se(2)–P(2)	2.1775(12)	Se(4)–P(4)	2.1874(12)
P(1)–N(1)	1.596(4)	P(3)–N(2)	1.584(4)
P(2)–N(1)	1.589(4)	P(4)–N(2)	1.583(4)
P(1)–C(11)	1.801(4)	P(3)–C(51)	1.814(5)
P(1)–C(21)	1.815(4)	P(3)–C(61)	1.819(4)
P(2)–C(31)	1.806(4)	P(4)–C(71)	1.811(4)
P(2)–C(41)	1.820(4)	P(4)–C(81)	1.814(4)
Se(1)–Te(1)–Se(2)	87.01(3)	Se(3)–Te(2)–Se(4)	86.43(3)
Se(1)–Te(1)–Se(2a)	92.99(3)	Se(3)–Te(2)–Se(4a)	93.57(2)
P(1)–Se(1)–Te(1)	95.99(4)	P(3)–Se(3)–Te(2)	87.37(5)
P(2)–Se(2)–Te(1)	94.82(4)	P(4)–Se(4)–Te(2)	95.03(4)
Se(1)–P(1)–N(1)	118.41(14)	Se(3)–P(3)–N(2)	118.78(15)
Se(2)–P(2)–N(1)	119.50(14)	Se(4)–P(4)–N(2)	119.26(14)
P(1)–N(1)–P(2)	137.2(2)|	P(3)–N(2)–P(4)	140.8(2)
Se(1)–P(1)–C(11)	103.6(2)	Se(3)–P(3)–C(51)	106.5(2)
Se(1)–P(1)–C(21)	110.8(2)	Se(3)–P(3)–C(61)	109.0(2)
N(1)–P(1)–C(11)	107.5(2)	N(2)–P(3)–C(51)	108.0(2)
N(1)–P(1)–C(21)	110.6(2)	N(2)–P(3)–C(61)	106.8(2)
C(11)–P(1)–C(21)	104.8(2)	C(51)–P(3)–C(61)	107.2(2)
Se(2)–P(2)–C(31)	104.6(2)	Se(4)–P(4)–C(71)	109.9(2)
Se(2)–P(2)–C(41)	109.9(2)	Se(4)–P(4)–C(81)	106.8(2)
N(1)–P(2)–C(31)	106.4(2)	N(2)–P(4)–C(71)	104.6(2)
N(1)–P(2)–C(41)	110.7(2)	N(2)–P(4)–C(81)	109.5(2)
C(31)–P(2)–C(41)	104.5(2)	C(71)–P(4)–C(81)	106.1(2)

TABLE 4 Selected Bond Lengths/Å and Angles/° for **2**

Te(1)–S(1)	2.551(3)	P(2)–N(1)	1.624(9)
Te(1)–S(2a)	2.873(3)	P(2)–C(20)	1.787(10)
Te(1)–C(1)	2.100(11)	P(2)–C(26)	1.802(9)
P(1)–N(1)	1.557(8)	P(2)–S(2)	2.006(4)
P(1)–C(14)	1.781(9)	O(1)–C(4)	1.381(12)
P(1)–C(8)	1.807(11)	O(1)–C(7)	1.41(2)
P(1)–S(1)	2.041(3)		
C(1)–Te(1)–S(1)	90.3(2)	C(20)–P(2)–C(26)	106.7(5)
C(1)–Te(1)–S(2a)	85.5(2)	N(1)–P(2)–S(2)	117.9(3)
S(1)–Te(1)–S(2a)	174.20(8)	C(20)–P(2)–S(2)	110.7(3)
N(1)–P(1)–C(14)	112.4(4)	C(26)-P(2)-S(2)	108.4(4)
N(1)-P(1)-C(8)	105.9(5)	P(1)–S(1)-Te(1)	98.42(12)
C(14)–P(1)–C(8)	106.5(5)	P(2)–S(2)–Te(1a)	109.71(13)
N(1)–P(1)–S(1)	118.2(3)	C(4)–O(1)–C(7)	116.6(9)
C(14)–P(1)–S(1)	105.0(4)	P(1)–N(1)–P(2)	132.1(6)
C(8)–P(1)–S(1)	108.3(3)	C(6)–C(1)–Te(1)	121.9(8)
N(1)–P(2)–C(20)	103.4(5)	C(2)–C(1)–Te(1)	120.0(7)
N(1)–P(2)–C(26)	109.2(4)		

earlier for Te(II) [9]. The average Te–S bond length is 2.712 Å, 0.30 Å longer than the sum of the covalent radii of S and Te [18]. This is 0.03 Å longer than the typical Te–S bond length in more symmetric three-center, four-electron systems centered on Te(II). This is probably due to the great asymmetry of the two Te–S bonds [9]. Another such 12-membered ring compound, **4,** has been prepared earlier by a different procedure [24]. The only difference from **2** is that **4** has a phenyl group bonded to Te instead of a methoxyphenyl group. The structures of **2** and **4** are quite similar, and the Te-ligand bond lengths in **4** are 2.109 (9), 2.557 (3), 2.843 (3), and 3.775 (1) Å for Te(1)–C(1), Te(1)–S(1), Te(1)–S(2a), and Te(1)–Te(1a), respectively.

The Ligands $Ph_2P(E)NP(E)Ph_2$, $E = Se,S$

These anionic ligands have their charges delocalized over the whole EPNPE skeleton. In **1**, with essentially isobidentate ligands, the average P$\overset{\cdots}{-}$Se bond length is 2.178 (1) Å, while the average P$\overset{\cdots}{-}$N bond length is 1.588 (6) Å. This may be compared to bond lengths of the free acid (proton on N) where the average P = Se bond length is 2.093 (8) and the average P–N bond length is 1.682 (4) Å [25]. Metal complexes with this ligand have similar bond lengths (P$\overset{\cdots}{-}$Se close to 2.18 Å, P$\overset{\cdots}{-}$N close to 1.58 Å) [6,7,25]. In **2,** the thio ligands are anisobidentate. This is reflected in the ligand bonds P(1)$\overset{\cdots}{-}$S(1) = 2.041 (3),

TABLE 5 Crystal Data and Structure Refinement Parameters

	1	**2** · *3CH$_2$Cl$_2$*
Empirical formula	$C_{48}H_{40}N_2P_4Se_4Te$	$(C_{65}H_{60}Cl_6N_2O_2P_4S_4Te_2)_{1/2}$
M	1212.14	1621.16
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a/Å	10.017(2)	11.390(2)
b/Å	13.117(4)	11.489(2)
c/Å	18.169(5)	14.893(3)
α/°	90.16(2)	90.24(2)
β/°	97.46(2)	103.51(2)
γ/°	102.07(2)	116.45(3)
Temp./K	103(2)	153(2)
V/Å^3	2313.7(10)	1683.4(5)
Z	2	1
Wave length/Å	0.71069	0.71069
D_c/mg·m^{-3}	1.740	1.599
μ/mm^{-1}	3.964	1.369
F(000)	1180	810
Crystal size (mm)	0.04 × 0.08 × 0.12	0.50 × 0.30 × 0.20
θ range/°	2.1–28.0	1.42–23.89
hkl ranges	−11/10, −17/17, 0/23	−10/11, −12/12, −16/0
Total no. of reflections	9150	4549
Crystal decay correction/%	max 1.8	None
Transmission coeff.	0.640–0.862	0.43–0.77
Independent reflections $I > 2\sigma(I)$ (R_{int})	7391	4549
Data (all)/parameters	9124/535	4383/470
Goodness of fit on F^2	1.043	1.034
Final R indices: R_1 (obs/all data)	0.0354/0.0554	0.0592/0.0817
wR_2 (obs/all data)	0.0827/0.1025	0.1762/0.1954
Largest difference peak and hole/e·Å^{-3}	1.014, −0.670	1.632, −1.322

P(1)$\overset{\cdots}{-}$S(2) = 2.006 (3), P(1)$\overset{\cdots}{-}$N(1) = 1.557 (8), and P(2)$\overset{\cdots}{-}$N(1) = 1.624 (9) Å. The corresponding bond lengths in the analogous compound, **4**, are 2.044 (4), 1.996 (4), 1.580 (7), and 1.589 (7) Å [24]. Delocalization of charge in the ligands is therefore uneven; a strong Te–S bond corresponds to a weaker S–P bond that again may correspond to a stronger P–N bond, and vice versa. The protonated thio ligand has average P=S and P–N bonds of 1.944 (7) and 1.678 (6) Å [26], while the free anionic ligand with a delocalized charge has P$\overset{\cdots}{-}$S and P$\overset{\cdots}{-}$N bond lengths of ca. 1.98 and 1.60 Å, respectively [27].

It is typical for $R_2P(E)NP(E)R_2'^{-}$ ligands (R = R′ = Ph, Me, *i*Pr; R = Ph, R′ = Me, E = O, S, Se) that the donor atoms (E) obtain a *syn* position relative to each other upon complex formation. The structures of the free and also of the protonated ligands (protonated at N except for E = O where one oxygen is protonated) has so far shown the E atoms *anti* to each other [5,27,28]. The P-N-P angle varies widely, reflecting the ligand bite flexibility. In **1**, the P-N-P angle of molecule A is 137.2 (2)° while that of B is 140.8 (2)°. This latter value seems to be a maximum, a little larger than that for **3** [1]. The P-N-P angles for **2** and **4** are 132.1(6) and 134.1(5)°. The minimum value of 120.4° is found in [Ni{(Ph$_2$P(S)NP(S)Me)$_2$}$_2$] [4]. In the ligand anion of the parent acid, the angle is around 132°; however, the anion $Ph_2P(S)NP(S)Ph_2^-$ has a linear P-N-P sequence in its salt with the $(Ph_3P\text{-}N\text{-}PPh_3)^+$ cation [27]. Surprisingly, the angular variation does not influence the P–N bond lengths in a systematic way, except for the linear anion above where the P–N bond length is only 1.554 (2). There the N atom is probably *sp* hybridized compared to more or less *sp*^{2} hybridization for the others, resulting in a P–N bond contraction of ca. 0.05 Å.

EXPERIMENTAL

Synthesis of [Te{(SePPh$_2$)$_2$N}$_2$]

The potassium salt of the ligand, K[(SePPh$_2$)$_2$N], was prepared by a published method [25]. To 1.6 g (2.0 mmol) of K[(SePPh$_2$)$_2$N] dissolved in 30 mL methanol was added under stirring a yellow solution of 0.54 g (1.0 mmol) $[Te(tu)_4]Cl_2 \cdot 2H_2O$ [29] (tu = thiourea) in 10 mL methanol. A yellow precipitate formed and was filtered off, washed with 2 × 10 mL methanol and 1 × 10 mL diethyl ether and dried in vacuum. Yield: 0.89 g (73.9%); Mp: 275°C. Calcd for $C_{48}H_{40}N_2P_4Se_4Te$: C, 47.76; H, 3.30; N, 2.31%. Found: C, 47.76; H, 3.23; N, 2.42%. $^{31}P\text{-}\{^1H\}$ NMR in $CH_2Cl_2/CDCl_3$: singlet $\delta = 24.4$ ppm, $^1J_{P\text{-}Se} = 586.4$ Hz.

Synthesis of [4-MeOPhTe{(SPPh$_2$)$_2$N}]$_2$

A mixture of 0.45 g (1.0 mmol) of $(SPPh_2)_2NH$ made by a literature method [30] and 0.112 g (1.0 mmol) of Me_3COK in 15 mL dry methanol was stirred in a beaker. To the resulting clear solution was added a yellow solution of 0.34 g (1.0 mmol) of 4-MeOPhTeCl$_3$ [31] in 10 mL methanol under stirring for 14 hours. The yellow precipitate was filtered off, washed with 2 × 10 mL methanol and 1 × 10 mL diethyl ether and dried in vacuum. Yield (after recrystallization from CH_2Cl_2 that resulted in $\mathbf{2} \cdot 3CH_2Cl_2$): 0.15 g (18.6%); Mp: 167°C. Calcd for $C_{62}H_{54}N_2O_2P_4S_4Te_2$: C, 54.49; H, 3.95; N, 2.05; S, 9.37%. Found: C, 54.21; H, 4.01; N, 1.91; S, 8.73%. $^{31}P\text{-}\{^1H\}$NMR (in $CH_2Cl_2/CDCl_3$): singlet $\delta = 34.9$ ppm.

Structure Determination

Crystal data and structure refinement parameters for **1** and **2** are shown in Table 5. Upon recrystallization, compound **2** picked up three solvent molecules (CH_2Cl_2). Cell dimensions were obtained from several carefully centered reflections (24 for **1** and 27 for $\mathbf{2} \cdot 3CH_2Cl_2$), using a least-squares procedure. Three standard reflections were measured at the following intervals: every 2 hours for **1**, and every 200 reflections for $\mathbf{2} \cdot 3CH_2Cl_2$. The intensity data were corrected for Lorentz and polarization effects, and for absorption [32]. All atoms, except hydrogens and disordered C(102) in **2**, were refined anisotropically. Hydrogen atoms were refined isotropically; for **1**, the isotropic temperature factors were constrained to values 1.5 times those of the connected carbon atoms. One CH_2Cl_2 molecule is disordered. Data collection for **1** was made on an Enraf-Nonius CAD-4 diffractometer and for **2** on a Kuma KM4 diffractometer. Both structures were solved and refined using the SHELXS86 and SHELXL93 programs [33,34].

SUPPLEMENTARY MATERIAL

Further details of the structural work is deposited with the Cambridge Crystallographic Data Centre and comprises atomic coordinates (including H), tables of bond distances and angles, and anisotropic temperature factors.

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