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TRIPHENYLTELLURONIUM DERIVATIVES OF DITHIOPHOSPHORUS LIGANDS: CRYSTAL AND MOLECULAR STRUCTURE OF $[Ph_3Te][S_2PPh_2]$ AND $[Ph_3Te][(SPPh_2)_2N]$, DISPLAYING WEAK CATION–ANION Te···S SECONDARY INTERACTIONS

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Abstract—Triphenyltelluronium diphenylphosphinodithioate, $[Ph_3Te][S_2PPh_2]$ (**1**) and tetraphenyldithioimidodiphosphinate, $[Ph_3Te][(SPPh_2)_2N]$ (**2**), were obtained by metathesis reaction between Ph_3TeCl and $NH_4[S_2PPh_2]$ and $K[(SPPh_2)_2N]$, respectively, and were characterized by IR, MS and multinuclear NMR. The molecular structure of both compounds was investigated by X-ray diffractometry. The compounds are monomeric and predominantly ionic in the solid state. The Te···S interatomic distances (3.331 and 3.655 Å in **1**, and 3.264 and 3.451 Å in **2** indicate weak cation–anion secondary interactions. In both compounds the tellurium atom is five-coordinated, with distorted square-pyramidal geometry. The coordination polyhedron in **2** is less distorted due to the flexibility of the SPNPS system and the larger S···S bite of this ligand (4.276 Å in **2**, vs 3.368 Å in **1**).

Triorganotelluronium salts, R_3TeX, of simple anions such as NCO^-,[1] NCS^-,[2] or Cl^-,[3] display rather complex structures due to secondary bonding interactions between the tellurium atom and the anion. This behaviour is favoured by the tendency of tellurium to achieve higher coordination numbers. With 1,1-dithio ligands either dimeric $[Ph_3TeS_2CNPr^i_2]_2$,[4] $[Ph_3TeS_2COMe]_2$,[5] and $[Ph_3TeS_2COEt]_2$[6] (bridging ligand) or monomeric $Ph_3TeS_2COBu^i$,[5] are formed. The use of dithiophosphorus ligands, of known ability to exhibit various coordination patterns,[7] might result in further knowledge concerning stereochemical aspects of tellurium complexes with sulphur ligands.[8] The synthesis and spectroscopic characterization of

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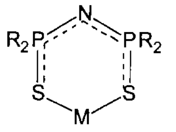
several triorganotelluronium derivatives of 1,1-dithiophosphorus ligands, $[R_3Te][S_2PR'_2]$ and $[R_3Te][S_2P(OR')_2]$,[9-11] as well as the X-ray structure determination of the monomeric $[Ph_3Te][S_2P(OEt)_2]$,[12] has been recently reported. In complexes containing 1,1-dithio ligands the coordination polyhedron of tellurium is generally strongly distorted due to the small bite ($S \cdots S$ *ca* 3.0 Å) of the ligand. Therefore, it would be of interest to compare the structures of compounds containing dithiophosphorus ligands with significantly differing $S \cdots S$ bites. Thus, tetraphenyldithioimidodiphosphinato ligand, $[(SPPh_2)_2N]^-$, which can form six-membered rings (**a**), will impose lesser steric constraints than the phosphinodithioates, which form four-membered rings (**b**) in monomeric compounds. The dimerization of **b** is a way to avoid this steric strain. So far, only Te^{II} complexes of dithioimidodiphosphinato ligand, i.e. $Te[(SPPh_2)_2N]_2$ (monomeric)[13] and $[PhTe(SPPh_2)_2N]_2$ (dimeric),[14] have been described.



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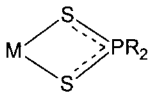


b

We report here the synthesis, spectroscopic characterization and X-ray molecular structure of $[Ph_3Te][S_2PPh_2]$ (**1**) and $[Ph_3Te][(SPPh_2)_2N]$ (**2**); both of which are monomeric and display weak $Te \cdots S$ secondary interactions between the triphenyltelluronium cation and the dithiophosphorus anion.

EXPERIMENTAL

The starting materials were prepared according to literature methods: Ph_3TeCl,[15] $NH_4[S_2PPh_2]$,[16] and $K[(SPPh_2)_2N]$.[17] Elemental analyses (carbon and hydrogen) were performed by Beller Mikroanalytisches Laboratorium (Göttingen). IR spectra were recorded in the range 4000–400 cm^{-1} as nujol mulls using a Bio-Rad-FTS7 instrument. ^1H, ^{13}C and ^{31}P NMR were obtained in $CDCl_3$ solutions on a Bruker WH 360 instrument operating at 360.134, 145.785 and 90.554 MHz, respectively. Mass spectra were recorded on Varian CH7 (A) (MAT) and Jeol SX-102A spectrometers using both EI and FAB(+) techniques.

Preparation of triphenyltelluronium diphenylphosphinodithioate, $[Ph_3Te][S_2PPh_2]$

A mixture of Ph_3TeCl (0.65 g, 1.65 mmol) and $NH_4[S_2PPh_2]$ (0.44 g, 1.65 mmol) in 25 cm^3 methanol was stirred at room temperature for 0.5 h. The resulting ammonium chloride was filtered off and the clear filtrate was evaporated in a rotary evaporator. The white solid product was recrystallized from chloroform. Yield: 0.86 g (85%); m.p. = 178°C [lit. 136°C (dec.)[11])]. Found: C, 59.3; H, 4.3. Calc. for $C_{30}H_{25}PS_2Te$: C, 59.2; H, 4.1. 1R (cm^{-1}): $\nu_{as}(PS_2)$ 645m, 620s; $\nu_s(PS_2)$ 570s, 535m. ^1H NMR (δ, relative to TMS): 7.23m (6H, P-C_6H_5, *meta*+*para*), 7.36m (6H, Te-C_6H_5, *meta*), 7.45m (3H, Te-C_6H_5, *para*), 7.66m (6H, Te-C_6H_5, *ortho*), 7.92ddd (4H, P-C_6H_5, *ortho*, $^3J_{PH}$ 13.9, $^3J_{HH}$ 6.7, $^{43}J_{HH}$ 3.0). ^{31}P NMR (δ, relative to H_3PO_4 85%): 59.6s (lit. 58.8 ppm[11]). MS-EI [70 eV; *m*/*z* (%)]: 361 (27), Ph_3Te^+; 284 (63), $PhTe^+$; 249 (11), $Ph_2PS_2{}^+$; 217 (24), Ph_2PS^+; 207 (21) $PhTe^+$;172 (100), $PhPS_2{}^+$; 154 (85), Ph_2^+. MS-FAB(+) [*m*/*z*, (%)]: 610 (1), M^+; 456 (1), $PhTeS_2PPh_2^+$; 361 (43), Ph_3Te^+; 284 (5), Ph_2Te^+; 249 (20), $Ph_2PS_2^+$; 217 (100), Ph_2PS^+; 207 (2) $PhTe^+$; 172 (1), $PhPS_2^+$; 154 (56), Ph_2^+.

Preparation of triphenyltelluronium tetraphenyldithioimidodiphosphinate, $[Ph_3Te][(SPPh_2)_2N]$

A mixture of Ph_3TeCl (0.27 g, 1.013 mmol) and $K[(SPPh_2)_2N]$ (0.49 g, 1.013 mmol) in 25 cm^3 methanol was stirred at room temperature for 2 h. The resulting KCl was filtered off and the solvent was removed from the clear solution in a rotary evaporator. The white solid product was recrystallized from acetone. Yield: 0.69 g (86%); m.p. = 195–196°C. Found: C, 62.2, H, 4.5. Calc. for $C_{42}H_{35}NP_2S_2Te$: C, 62.5; H, 4.49. 1R (cm^{-1}): $\nu(PS)$ 595s, 570m; $\nu_{as}(P_2N)$ 1204s, 1190s. ^1H NMR (δ, relative to TMS): 7.27m (18H, P-C_6H_5, *meta*+*para*, Te-C_6H_5, *meta*), 7.40m (3H, Te-C_6H_5, *para*), 7.48m (6H, Te-C_6H_5, *ortho*), 8.01ddd (8H, P-C_6H_5, *ortho*, $^3J_{PH}$ 13.3, $^3J_{HH}$ 7.7, $^4J_{HH}$ 1.7). ^{13}C NMR (δ, relative to TMS): 127.53m ($C_m(P)$, $^3J_{PC}$ 13.8), 128.88s ($C_i(Te)$), 129.37s ($C_p(Te)$), 129.93s ($C_m(Te)$), 130.97s ($C_p(P)$), 131.26m ($C_0(P)$, $^2J_{PC}$ 11.6), 135.00s ($C_0(Te)$), 142.16dd ($C_i(P)$, $^1J_{PC}$ 104.4, $^3J_{PC}$4.6). ^{31}P NMR (δ, relative to H_3PO_4 85%): 36.9s. MS-EI [70 eV; *m*/*z*, (%)]: 525 (28), $(SPPh_2)_2NPh^+$; 448 (9), $(SPPh_2)_2N^+$; 416 (100), $(SPPh_2)(PPh_2)N^+$; 361 (22), Ph_3Te^+; 284 (54), Ph_2Te^+; 217 (23), Ph_2PS^+; 185 (38), Ph_2P^+; 154 (87), Ph_2^+. MS-FAB(+) [*m*/*z*, (%)]: 809 (1), M^+; 525 (1), $(SPPh_2)_2NPh^+$; 448 (17), $(SPPh_2)_2N^+$; 416 (5), $(SPPh_2)(PPh_2)N^+$; 361 (100), Ph_3Te^+; 284 (4), Ph_2Te^+; 217 (8), Ph_2PS^+; 207 (3) $PhTe^+$; 185 (4), Ph_2P^+; 154 (15), Ph_2^+.

X-Ray crystal structure determination

Data were collected at room temperature on a Siemens P4 four-circle diffractometer with graphitemonochromated Mo-K_α radiation and the full-matrix least-squares refinement was performed with the SHELXTL-PC program system.[18] Details of crystal data, measurement of intensities and data processing are summarized in Table 1. The structures were solved by direct methods (SIR92).[19] All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were calculated as a riding model with fixed isotropic U.

RESULTS AND DISCUSSION

The title compounds were obtained by reacting Ph_3TeCl with the ammonium or potassium salt of the corresponding dithio ligand, in methanol (eqs 1 and 2):

$$Ph_3TeCl + NH_4[S_2PPh_2] \longrightarrow [Ph_3Te][S_2PPh_2] + NH_4Cl \quad (1)$$

$$Ph_3TeCl + K[(SPPh_2)_2N] \longrightarrow [Ph_3Te][(SPPh_2)_2N] + KCl. \quad (2)$$

After recrystallization from chloroform and acetone, respectively, both compounds were isolated as colourless crystals, soluble in methanol, chloroform and acetone, as well as water, thus suggesting a strong ionic character. The synthesis of $[Ph_3Te][S_2PPh_2]$ (from Ph_3TeCl and $Na[S_2PPh_2]$, or Ph_3TeOMe and HS_2PPh_2), as well as its ^1H and ^{31}P NMR spectra and conductivity data in MeCN were recently reported.[10,11]

The IR spectrum of $[Ph_3Te][S_2PPh_2]$ exhibits strong to medium absorption bands in the 650–550 cm^{-1} region, characteristic of asymmetric and symmetric phosphorus–sulphur stretching vibrations [$\nu_{as}(PS_2)$ 645m, 620s, and $\nu_s(PS_2)$ 570s, 535m]. These are close to those observed in the ammonion salt, $NH_4^+[S_2PPh_2]^-$ [$\nu_{as}(PS_2)$ 635vs, $\nu_s(PS_2)$ 557vs].

The IR spectrum of $[Ph_3Te][(SPPh_2)_2N]$ exhibits strong to medium absorption bands at 595s, 570m, and 1204s, 1290s cm^{-1}, respectively, which are indicative of the deprotonated ligand. These values clearly differ from those of the "free acid", $(SPPh_2)_2NH$ [ν(PS) 645vs; $\nu_{as}(P_2NH)$ 920vs], and are close to the values observed for the potassium salt, $K^+[(SPPh_2)_2N]^-$: ν(PS) 600vs, 580s; $\nu_{as}(P_2N)$ 1195vs, 1170vs.

The ^1H NMR spectra of both compounds exhibit the expected resonance signals for phenyl groups attached to tellurium and phosphorus, respectively. The ^{13}C NMR spectrum of $[Ph_3Te][SPPh_2)_2N]$

Table 1. Crystal data for $[Ph_3Te][S_2PPh_2]$ (**1**) and $[Ph_3Te][(SPPh_2)_2N]$ (**2**)

	1	**2**
Formula	$C_{30}H_{25}PS_2Te$	$C_{42}H_{35}NP_2S_2Te$
M	608.2	807.4
Crystal size (mm)	0.44 × 0.42 × 0.24	0.64 × 0.34 × 0.16
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
a (Å)	14.168(2)	12.440(2)
b (Å)	12.196(2)	19.615(4)
c (Å)	15.907(2)	15.798(3)
β (°)	103.22(2)	104.10(3)
V(Å^3)	2675.8(5)	3738.7(11)
Z	4	4
D_{calc} (g cm^{-3})	1.510	1.434
μ (Mo-K_α) (mm^{-1})	1.345	1.024
F(000)	1216	1632
Reflections collected	9378	6874
Independent reflections	4693 (R_{int} = 3.08%)	6550 (R_{int} = 3.39%)
Observed reflections	3646 ($F > 3.0\sigma(F)$)	3908 ($F > 3.0\sigma(F)$)
Absorption correction	Semi-empirical	Face-indexed numerical
Min./Max. transmission	0.7495/0.9431	0.7014/0.8506
Weighting scheme	$w^{-1} = \sigma^2(F) + 0.0002F^2$	$w^{-1} = \sigma^2(F) + 0.0008F^2$
R^a(%)	3.51	5.80
wR^a(%)	3.39	6.43
Goodness-of-fit	1.03	1.06

[a] $R = \Sigma|\Delta|/\Sigma|F_o|$; $wR = [\Sigma(w\Delta^2)/\Sigma(wF_o^2)]^{1/2}$; $S = [\Sigma w\Delta^2/(N.O. - N.V.)]^{1/2}$; $\Delta = |F_o - F_c|$.

Table 2. Interatomic distances (Å) and angles (°) in $[Ph_3Te][S_2PPh_2]$ (**1**)

Te—C(1)	2.129(4)	C(7)—Te—C(1)	101.2(2)
Te—C(7)	2.129(4)	C(7)—Te—C(13)	95.8(2)
Te—C(13)	2.139(4)	C(7)—Te···S(1)	90.2
		C(7)—Te···S(2)	90.5
Te···S(1)	3.331	C(1)—Te—C(13)	94.4(2)
Te···S(2)	3.655	C(13)—Te···S(2)	122.9
		S(2)···Te···S(1)	57.4
S(1)···S(2)	3.368	S(1)···Te—C(1)	83.8
		C(1)—Te···S(2)	139.7
		C(13)—Te···S(1)	174.0
		Te···S(1)—P	86.2
		Te···S(2)—P	77.9
P—S(1)	2.004(2)	S(1)—P—S(2)	115.7(1)
P—S(2)	1.975(2)	S(1)—P—C(19)	108.7(1)
P—C(19)	1.836(4)	S(1)—P—C(25)	109.5(2)
P—C(25)	1.833(4)	S(2)—P—C(19)	109.6(2)
		S(2)—P—C(25)	110.6(1)
		C(19)—P—C(25)	101.7(1)

shows, as expected, a splitting of the signals for carbon atoms of phenyl groups bound to phosphorus, due to phosphorus–carbon spin couplings.

Both compounds exhibit only one sharp, singlet ^{31}P resonance, which indicates equivalence of the two phosphorus atoms in the ligand unit of $[Ph_3Te][(SPPh_2)_2N]$ in solution.

The solid state molecular structure of the title compounds was investigated by X-ray diffractometry. Selected interatomic distances and angles are listed in Tables 2 and 3, while ORTEP-like diagrams of the molecular structures, with the atom numbering schemes, are shown in Fig. 1 for $[Ph_3Te][S_2PPh_2]$ (**1**), and Fig. 2 for $[Ph_3Te][(SPPh_2)_2N]$ (**2**).

Both compounds are monomeric and contain a pyramidal Ph_3Te unit, with C—Te—C angles averaging 97.1° for **1**, and 97.3° for **2**. The mean Te—C bond distances are 2.132 Å for **1**, and 2.131 Å for **2**. These values are very close to those observed in the dimeric Ph_3TeCl used as starting material (C—Te—C av. 95.1° and Te—C av. 2.130 Å, respectively).[3]

The phosphinodithioato ligand in $[Ph_3Te][S_2PPh_2]$ exhibits slightly non-equivalent phos-

Table 3. Interatomic distances (Å) and angles (°) in $[Ph_3Te][(SPPh_2)_2N]$ (**2**)

Te—C(1)	2.129(9)	C(13)—Te—C(1)	97.4(4)
Te—C(7)	2.143(8)	C(13)—Te—C(7)	97.3(3)
Te—C(13)	2.121(9)	C(13)—Te···S(1)	89.4
		C(13)—Te···S(2)	85.9
Te···S(1)	3.264	C(1)—Te—C(7)	96.8(3)
Te···S(2)	3.451	C(7)—Te···S(2)	94.5
		S(2)···Te···S(1)	79.1
S(1)···S(2)	4.276	S(1)···Te—C(1)	89.0
		C(1)—Te···S(2)	167.7
		C(7)—Te···S(1)	170.5
		Te···S(1)—P(1)	86.5
		Te···S(2)—P(2)	101.1
P(1)—S(1)	1.983(4)	S(1)—P(1)—N	120.1(4)
P(2)—S(2)	1.974(5)	S(2)—P(2)—N	120.9(4)
P(1)—N	1.594(9)	P(1)—N—P(2)	137.6(6)
P(2)—N	1.580(9)		

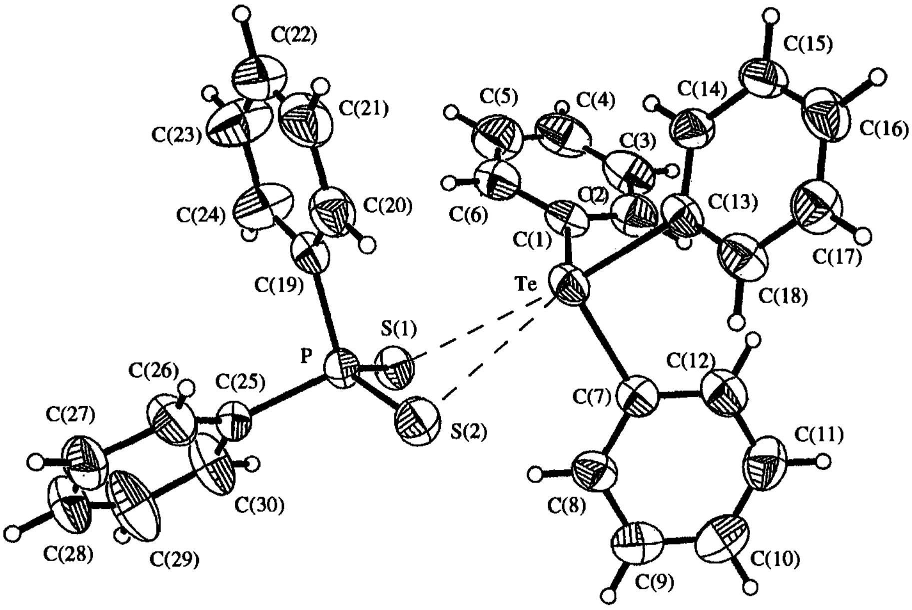


Fig. 1. ORTEP-like view of the $[Ph_3Te][S_2PPh_2]$ molecule (**1**).

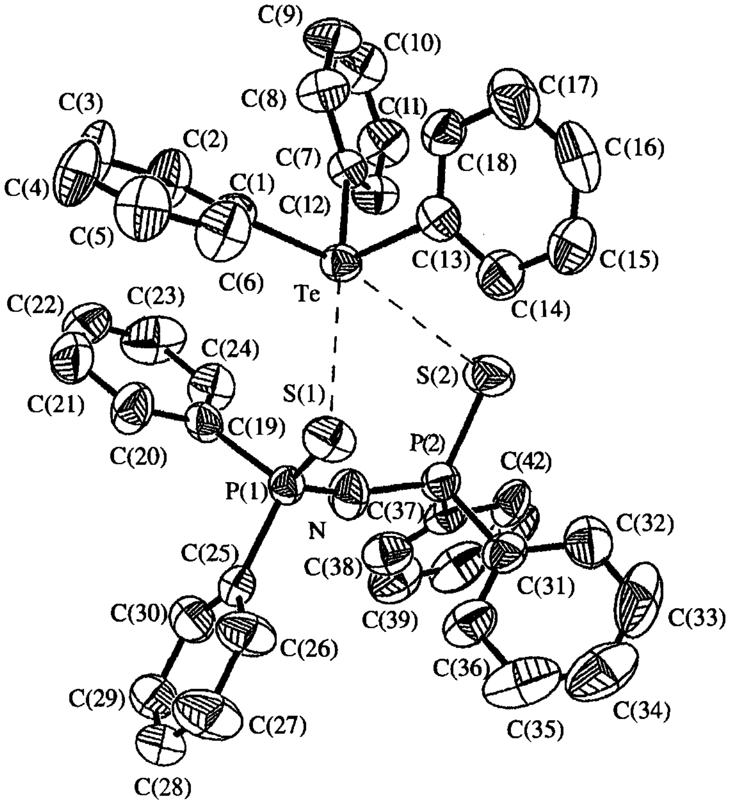
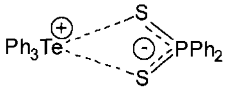


Fig. 2. ORTEP-like view of the $[Ph_3Te][(SPPh_2)_2N]$ molecule (**2**).

phorus–sulphur distances [P—S(1) 2.004(2) Å and P—S(2) 1.975(2) Å, vs 2.077(1) and 1.954(1) Å in the free $Ph_2P(S)SH$ acid.[20]] This suggests a high degree of delocalization of the π-electrons over the PS_2 system, however non-symmetrical. By contrast, in $[Ph_3Te][(SPPh_2)_2N]$ the dithioimidodiphosphinato ligand has equal phosphorus–sulphur [av. 1.979(5) Å] and phosphorus–nitrogen [av.

1.587(9) Å] bonds, consistent with a basically symmetrical distribution of the π-electrons. The P—S distances in the SPNPS fragment are close to those observed in the free anion of [K(18-crown-6)][$(SPPh_2)_2N$] [1.976(1) Å],[21] but longer than in the "free acid" $(SPPh_2)_2NH$ [1.937(1) and 1.950(1) Å][22] and shorter than in the Te[$(SPPh_2)_2N$]$_2$ chelate [av. 2.022(3) Å].[13]

In complexes **1** and **2** weak tellurium–sulphur secondary interactions [Σ_{vdw}(Te,S) = 3.9 Å][23] are established between the two sulphur atoms of the ligand fragment and the tellurium atom (Tables 2 and 3). In the dithiophosphato analogue, [Ph_3Te] [$S_2P(OEt)_2$],[12] the Te···S distances are slightly shorter, i.e. 3.150 and 3.286 Å. Both interatomic Te···S distances for a molecular unit are larger than the sum of the corresponding covalent radii [Σ_{cov}(Te,S) = 2.37 Å][23] or the Te—S distances in the Te[$(SPPh_2)_2N$]$_2$ chelate (av. 2.685(2) Å).[13] The elongation of the tellurium–sulphur bonds may be due to the strong *trans* effect of the phenyl groups, also observed in other organotellurium derivatives of dithio ligands.[8] As a result the chelate rings normally formed by coordination of the bidentate dithiophosphorus ligand to a metal, in this case become a cation–anion association through Te···S secondary bonds, additional to electrostatic interactions [structures **c** and **d**]. Such Te···S secondary interactions are usually observed as intermolecular bonds in associated tellurium–sulphur derivatives, e.g. [$ArTeS_2P(OMe)_2$]$_n$,[24,25] and [$PhTeS_2PPh_2$]$_n$.[26,27] The coordination pattern of the dithio moiety can be described as monometallic biconnective.[7]



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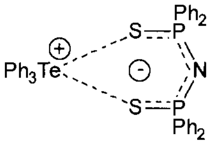

d

In the case of compound **2** the bidentate nature of the ligand is substantiated by the relative position of the sulphur atoms with respect to the PNP fragment. In the "free acid" $(SPPh_2)_2NH$,[22] the S—P—N—P—S backbone has an *anti* conformation, with a long sulphur–sulphur (non-interacting) distance of 5.708 Å. In the title compound the S—P—N—P—S fragment is twisted to achieve a *syn* conformation, which brings the sulphur atoms much closer (S···S 4.276 Å) and allows interactions of both sulphur atoms with tellurium.

As a consequence of the weak Te···S interactions, in both compounds the tellurium atom is

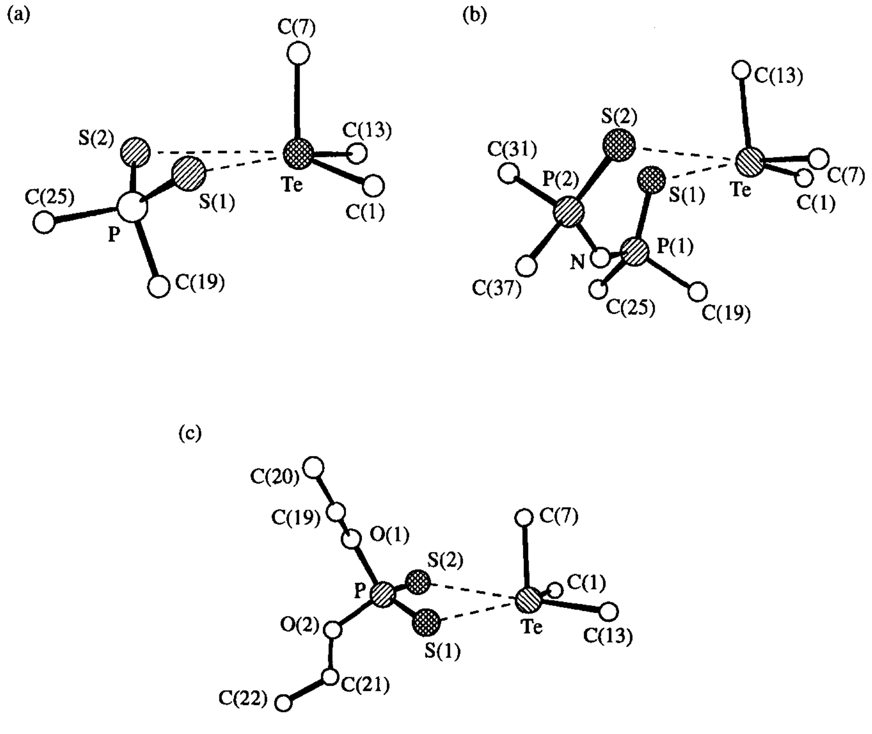

Fig. 3. Coordination geometry and chelate ring conformation in (a) [Ph_3Te][S_2PPh_2] (**1**), (b) [Ph_3Te] [$(SPPh_2)_2N$] (**2**), and (c) [Ph_3Te][$S_2P(OEt)_2$][12] (for clarity only *ipso* carbons of phenyl groups are shown).

five-coordinated in a distorted square-pyramidal geometry (see angles at Te in Tables 2 and 3). The apical position is occupied by C(7) in **1** and C(13) in **2**, respectively. The deviations from the best basal plane are: C(1)C(13)S(1)S(2) plane – C(1) −0.093, C(13) 0.069, S(1) 0.087, S(2) −0.063, Te 0.210, C(7) (*apical*) 2.335, P −0.889 Å for **1**, and C(1)C(7)S(1)S(2) plane – C(1) 0.044, C(7) −0.041, S(1) −0.033, S(2) 0.030, Te 0.126, C(13) (*apical*) 2.209, P(1) −1.877, P(2) −1.232, N −2.113 Å for **2**. In both cases the tellurium atom is slightly displaced towards the apical atom, and the four-membered TeS_2P and six-membered TeS_2P_2N rings are folded in opposite directions [dihedral angles: TeS(1)S(2)/S(1)S(2)P 125.1°, and TeS(1)S(2)/S(1)S(2)P(1)P(2)N 114.4°]. This contrasts with the situation observed in $[Ph_3Te][S_2P(OEt)_2]$,[12] for which the four-membered TeS_2P ring is almost planar (dihedral angle TeS(1)S(2)/S(1)S(2)P 9.2°). The deviation of the C(13)—Te···S(2) (122.9°) and S(1)···Te···S(2) (57.4°) basal angles in **1** from the ideal value of 90° is much larger than the corresponding C(7)—Te···S(2) (94.5°) and S(1)···Te···S(2) (79.1°) angles in **2**. This is caused by the smaller S(1)···S(2) ligand bite in **1** (3.368 Å) than in **2** (4.276 Å). Similar values for the corresponding angles were observed in $[Ph_3Te][S_2P(OEt)_2]$,[12] i.e. C(13)—Te···S(1) 109.9(2)° and S(1)···Te···S(2) 62.74(7)° for a S···S bite of 3.354 Å. The coordination geometries and ring conformations for $[Ph_3Te][S_2PPh_2]$, $[Ph_3Te][(SPPh_2)_2N]$ and $[Ph_3Te][S_2P(OEt)_2]$ are presented for comparison in Fig. 3.

Alternatively, the coordination geometry at tellurium might be described as *pseudo*-octahedral, with an electron lone pair of tellurium *trans* to the apical position occupied by the carbon atom.

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REFERENCES

1. D. D. Titus, J.-S. Lee and R. F. Ziolo, *J. Organomet. Chem.* 1976, **120**, 381.
2. J.-S. Lee, D. D. Titus and R. F. Ziolo, *Inorg. Chem.* 1977, **16**, 2487.
3. R. F. Ziolo and M. Extine, *Inorg. Chem.* 1980, **19**, 2964.
4. J. E. Drake and M. L. Y. Wong, *J. Organomet. Chem.* 1989, **377**, 43.
5. A. K. Singh, J. K. Basumatary, T. P. Singh and B. Padmanabhan, *J. Organomet. Chem.* 1992, **424**, 33.
6. V. Srivastava, J. K. Basumatary, A. K. Singh, T. P. Singh and A. K. Saxena, *Phosphorus Sulfur* 1993, **85**, 175.
7. I. Haiduc, *Rev. Inorg. Chem.* 1981, **3**, 353.
8. I. Haiduc, R. B. King and M. G. Newton, *Chem. Rev.* 1994, **94**, 301.
9. A. K. Singh and J. K. Basumatary, *J. Organomet. Chem.* 1989, **364**, 73.
10. M. Wieber and S. Rohse, *Phosphorus Sulfur* 1991, **55**, 85.
11. M. Wieber and S. Rohse, *Phosphorus Sulfur* 1991, **55**, 91.
12. M. Wieber, S. Lang and N. Graf, *Phosphorus Sulfur* 1993, **85**, 31.
13. S. Bjornevag, S. Husebye and K. Maartmann-Moe, *Acta Chem. Scand.* 1982, **A36**, 195.
14. S. Husebye, K. Maartmann-Moe and O. Mikalsen, *Acta Chem. Scand.* 1990, **44**, 802.
15. W. H. H. Guenther, J. Nepywoda and J. Y. C. Chu, *J. Organomet. Chem.* 1974, **74**, 79.
16. C. Silvestru, A. Silvestru, I. Haiduc, R. Gaviño-Ramirez and R. Cea-Olivares, *Heteroatom Chem.* 1994, **15**, 327.
17. R. Rösler, C. Silvestru, I. Haiduc, F. Kayser, M. Gielen, B. Mahieu, *Main Group Metal Chem.* 1993, **16**, 435.
18. G. M Sheldrick, SHELXTL-PC User's Manual, Siemens Analytical X-Ray Instruments, Inc., Madison, WI, (1990).
19. A. Altomare, G. Cascarano, C. Giacovazzo and A. Guagliardi, *J. Appl. Cryst.* 1993, **26**, 343.
20. B. Krebs and G. Henkel, *Z. Anorg. Allg. Chem.* 1981, **475**, 143.
21. R. Cea-Olivares and H. Nöth, *Z. Naturforsch.* 1987, **42b**, 1507.
22. S. Husebye and K. Maartmann-Moe, *Acta Chem. Scand.* 1983, **A37**, 439.
23. J. E. Huheey, *Inorganic Chemistry*, p. 278. Walter de Gruyter, Berlin (1988).
24. S. Husebye, K. Maartmann-Moe and O. Mikalsen, *Acta. Chem. Scand.* 1989, **3**, 868.
25. S. Husebye, K. Maartmann-Moe and O.Mikalsen, *Acta Chem. Scand.* 1990, **44**, 464.
26. A. Silvestru, I. Haiduc, K. H. Ebert and H. J. Breunig, *Inorg. Chem.* 1994, **33**, 1253.
27. A. Silvestru, I Haiduc, K. H. Ebert, H. J. Breunig and D. B. Sowerby, *J. Organomet. Chem.* 1994, **482**, 253.