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Synthesis and spectroscopic characterization of new mixed ligand organotellurium(IV) compounds employing dithiocarbamates and imidotetraphenyldithiodiphosphinates. Crystal structure of $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$, $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ and $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$

Graciela Canseco-Melchor *, Verónica García-Montalvo, R. Alfredo Toscano, Raymundo Cea-Olivares

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Mexico 04510 D.F., Mexico

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Abstract

The synthesis of new mixed ligand organotellurium(IV) compounds, $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$ (**1**), $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ (**2**), $[C_4H_8Te(S_2CNC_4H_8O)\{(SPPh_2)_2N\}]$ (**3**) and $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$ (**4**), was achieved. They were characterized by FAB$^+$ mass spectrometry, IR, ^{1}H-, ^{13}C-, ^{31}P- and ^{125}Te-NMR spectroscopy. The crystal structures of **1**, **2** and **4** were determined by X-ray diffraction. The solid state structures show that the coordination geometry can be described as the sawhorse structure typical for Te(IV) in which the lone pair is regarded as stereochemically active and occupying an equatorial position in a distorted trigonal bipyramid. Both types of ligands exhibit an anisobidentate chelating coordination mode on interaction with the tellurium center. If the aniso-bonded donor atoms are included in the coordination sphere, the coordination number increases to seven and the environment at Te can be described as the 1:2:2:2 structure. No intermolecular interaction was observed. © 2001 Published by Elsevier Science B.V.

Keywords: Tellurium; Dithiocarbamates; Imidodithiodiphosphinates; Metallacycles; Crystal structures

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1. Introduction

The importance of supramolecular associations in organotellurium compounds containing sulfur ligands has been pointed out in a recent review [1]. The ligands used include mono- and bidentate sulfur ligands such as *N*,*N*-dialkyldithiocarbamates [2–5], *O*-alkyl dithiocarbonates [5c,6], dithiophosphates [5,7], and tetraphenylimidodiphosphinates [8]. The bidentate ligands display unsymmetrical bidentate coordination (anisobidentate) on interacting with tellurium. In many of the compounds containing small bite ligands and/or halogen atoms, the tellurium atom exhibits a strong tendency to achieve higher coordination numbers through intra- or intermolecular Te···donor atom links. This and the apparently stereochemically active lone pair lead to a great structural diversity in the coordination geometry. On the other hand, few papers dealing with derivatives containing mixed ligands have appeared [9]. It seems that the use of two different types of ligand reduces the tendency for intermolecular association. However, the coordination polyhedra of tellurium are strongly distorted when 1,1-dithiolates are used, probably because of their relative short bite lengths. Given that the imidodithiodiphosphinates possess a wide degree of ring flexibility and relatively unrestricted bite lengths [8d,10], we decided to investigate the structure of new mixed organotellurium compounds using dithiocarbamates and the $[(SPPh_2)_2N]^-$ ligand.

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* Corresponding author. Fax: + 52-5-616-2203/17.

E-mail address: cea@servidor.unam.mx (G. Canseco-Melchor).

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In this study, we report the synthesis of four tellurole-1,1-diyl derivatives of the type [$C_4H_8Te(dtc)$-{$(SPPh_2)_2N$}] where dtc = S_2CNEt_2 (**1**), $S_2CNC_5H_{10}$ (**2**), $S_2CNC_4H_8O$ (**3**) and $S_2CNC_4H_8S$ (**4**). They were characterized by elemental analyses, positive ion FAB mass spectrometry, IR, and ^1H-, ^{13}C-, ^{31}P- and ^{125}Te-NMR spectroscopy. The crystal structures of **1**, **2**, and **4** have been determined by single-crystal X-ray diffraction analysis.

2. Results and discussion

Preparation of **1** to **4** was achieved by the reaction of an excess of the sodium salt of tetraphenyldithioimidodiphosphinate with $C_4H_8TeI(dtc)$ (dtc = S_2CNEt_2 **1**, $S_2CNC_5H_{10}$ **2**, $S_2CNC_4H_8O$ **3** and $S_2CNC_4H_8S$ **4**), in ethanol. All compounds were isolated by crystallization from CH_2Cl_2–*n*-hexane. They exhibit gradual decomposition in solution as has been observed for other sulfur-bonded organotellurium derivatives [5e,f,7,8].

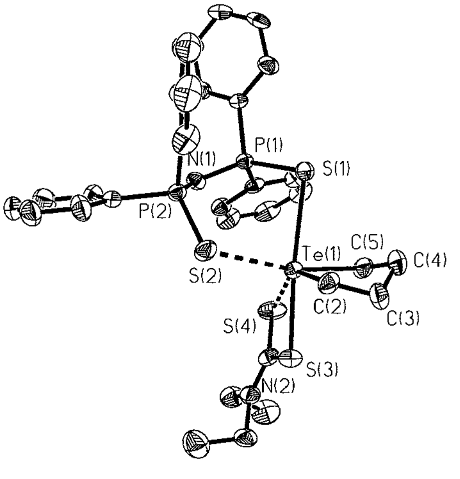

Fig. 1. Molecular structure of $C_4H_8Te(S_2CNEt_2)[(SPPh_2)_2N]$ (**1**). The ellipsoids enclose 30% probability.

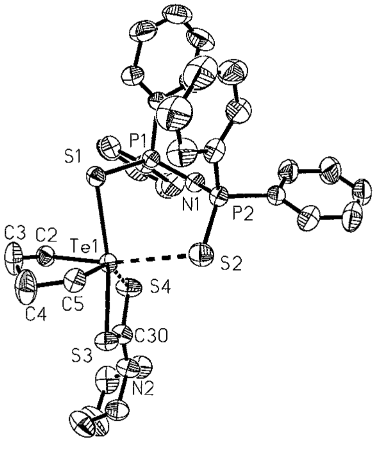

Fig. 2. Molecular structure of $C_4H_8Te(S_2CNC_5H_{10})[(SPPh_2)_2N]$ (**2**). The ellipsoids enclose 30% probability.

In their IR spectra the presence of two weak absorptions in the region 950–1050 cm^{-1}, characteristic of the ν(CS) vibration, suggests an anisobidentate behavior of the 1,1-dithiocarbamate ligands [11]. The $\nu(P_2N)$ vibrations characteristic for the tetraphenyldithiodiphosphinate as a free acid ligand and the anion $[(SPPh_2)_2N]^-$ are assigned to the bands at 920 and 781 cm^{-1} and 1199 and 808 cm^{-1}, respectively [12]. In our mixed compounds, these vibrations are closest to the values of the anion, indicating deprotonation of the ligand and the interaction of the anion with tellurium. The ν(PS) vibrations are observed in the range 600–500 cm^{-1}.

The positive ion FAB mass spectra show low intensity signals for the corresponding molecular ions. The most intense peak in all of the spectra corresponds to the $[(SPPh_2)_2N^+]$ (448 *m*/*z*) fragment. In addition, the spectra show some important fragments containing tellurium, e.g. [$^{130}Te\{(SPh_2)_2N\}^+$] (578 *m*/*z*) and [$C_4H_8^{130}Te^+$] (186 *m*/*z*). All of the observed signals have the characteristic isotopic distribution patterns.

The ^1H- and ^{13}C-NMR spectra show the expected signals for the organyltellurium group and the ligand organic groups. The phenyl carbon resonances exhibit coupling with ^{31}P. The ^{31}P spectra exhibit a single signal for the two equivalent phosphorus atoms. The chemical shifts are very similar in the range 36.43–36.50 ppm. These chemical shifts are far from the 56.87 ppm of the free acid ligand [13], but closer to that of the ligand potassium salt (37.1 ppm) [14] and to those of the related mixed compounds [9e]. The ^{125}Te signals are deshielded in comparison with that of $C_4H_8TeI_2$ (782 ppm in $CDCl_3$) and those of the analogous mixed compounds [9e], but are downfield shifted in comparison with $C_4H_8TeI[(SPPh_2)_2N]$ (833 ppm in $CDCl_3$) and $C_4H_8Te[(SPPh_2)_2N]_2$ (834.5 ppm in $CDCl_3$). All the spectra exhibit only small additional signals corresponding to [$Te\{(SPPh_2)_2N\}_2$] and $(Ph_2PS)_2NH$, indicating that the compounds undergo negligible dissociation and decomposition.

2.1. Description of the structures

Figs. 1–3 show the ORTEP diagrams with the atomic numbering scheme for [$C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}$] (**1**), [$C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}$] (**2**) and [$C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}$] (**4**), respectively.

The structures are constructed from discrete [$C_4H_8Te(dtc)\{(SPPh_2)_2N\}$] molecules (dtc is S_2CNEt_2 **1**, $S_2CNC_5H_{10}$ **2**, $S_2CNC_4H_8O$ **3**, and $S_2CNC_4H_8S$ **4**). The coordination environment can be described as the sawhorse structure typical of tellurium(IV) [1–8] in which the lone pair is assumed to be stereochemically active occupying an equatorial position in a trigonal bipyramid. The carbon atoms occupy the remaining equatorial positions (average Te–C 2.147 Å). Both types of ligands display an asymmetrical chelating coor-

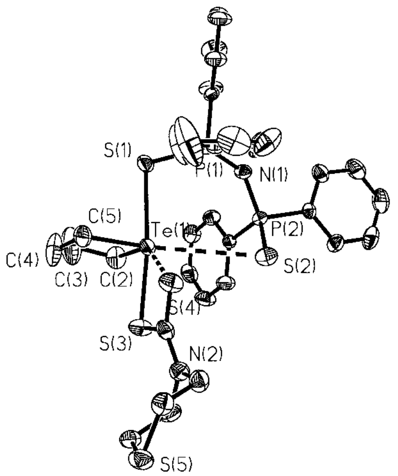

Fig. 3. Molecular structure of $C_4H_8Te(S_2CNC_4H_8S)[(SPPh_2)_2N]$ (**4**). The ellipsoids enclose 30% probability.

dination mode on interaction with the tellurium center, resulting in two distinct sets of Te–S bond lengths (Table 1). The two strongly bonded sulfur atoms occupy the axial positions. The average short Te–S bond distances are 2.810 Å for $[(Ph_2PS)_2N]^-$ and 2.532 Å for $[R_2NCS_2]^-$. These values are comparable with those reported for related structures containing analogous $[(R_2PS)_2N]^-$ [8a,d,15], and $R_2(S)P(CH_2)_2P(S)R_2$ [15a,b] ligands. The weakly bonded sulfur atoms are located at shorter Te···S distances than the sum of the van der Waals radii, 3.86 Å [16], with average Te···S lengths of 3.562 Å for $[(Ph_2PS)_2N]^-$ and 3.146 Å for $[R_2NCS_2]^-$. Although these interactions are larger than the axial Te–S bond lengths, they are still compatible with their being part of the coordination sphere and hence anisobonded. Therefore, the geometry at Te(IV) can be described as the 1:2:2:2 structure with a coordination number of seven [9e,17].

The average S–Te–S axial angles is 171.2°; this is larger than that found in the analogous mixed ligand compounds of 167.3° [9e], but similar to that of $[C_4H_8Te\{(Ph_2PS)_2N\}_2]$ (170.59(5)°) [8d].

As expected, the PNP unit in the tetraphenylimidodithiodiphosphinate ligands is angular. The P–N–P angles cover the range of 131.3(4) to 137.7(3)°. According to this, the resulting TeS_2P_2N rings display different conformations. The best description for the TeS_2P_2N arrangement in **1** and **2** is that of a twisted boat. The ring conformation of **4** is similar to the folded geometry observed in $[C_4H_8Te\{(Ph_2PS)_2N\}_2]$ [8d] and related mixed ligand compounds [9e], in which one phosphorus atom lies within the coordination plane and with the P–N–P–S plane folded by 65.2° out of the coordination plane. The torsion angles of the rings are listed in Table 2. These variations are consistent with the ring flexibility of the imidophosphinates [8d,10].

3. Experimental

3.1. General considerations

Chemicals of commercial grade were purchased from Aldrich and were used as supplied. The $C_4H_8TeI_2$ was prepared according to the method of Al-Rubaie [18], the 1,1-dithiocarbamate salts were obtained by procedures described in the literature [19]; $C_4H_8TeI(dtc)$ (dtc = S_2CNEt_2, $S_2CNC_5H_{10}$, $S_2CNC_4H_8O$ and $S_2CNC_4H_8S$) were prepared according to Dakternieks [20], and $Na[(SPPh_2)_2N]$ was prepared as described previously [21]. The IR spectra were recorded on a Nicolet FT-IR Magna 750 spectrometer (KBr disks) and Nicolet FT-IR (polyethylene disks). The FAB mass

Table 1
Bond distances (Å) and angles (°) with standard deviations of $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$ (**1**), $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ (**2**) and $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$ (**4**)

	1	**2**	**4**
Bond distances			
C2–C3	1.44(2)	1.508(10)	1.502(11)
C3–C4	1.54(3)	1.435(11)	1.425(14)
C4–C5	1.63(2)	1.536(11)	1.474(12)
N1–P1	1.590(5)	1.584(5)	1.570(4)
N1–P2	1.612(5)	1.604(5)	1.609(4)
S1–P1	2.025(2)	2.022(2)	2.0396(19)
S2–P2	1.980(2)	1.977(2)	1.9559(19)
S3–C30	1.782(7)	1.769(6)	1.765(6)
S4–C30	1.674(7)	1.690(6)	1.676(6)
Te1–C2	2.148(7)	2.151(6)	2.137(6)
Te1–C5	2.146(6)	2.151(6)	2.141(6)
Te1–S1	2.842(2)	2.817(2)	2.7736(15)
Te1···S2	3.367(2)	3.616(2)	3.7035(18)
Te1–S3	2.534(2)	2.523(2)	2.5392(16)
Te1···S4	3.180(2)	3.099(2)	3.1619(18)
Bond angles			
C5–Te1–C2	83.8(3)	84.5(2)	84.0(3)
N1–P1–S1	117.7(2)	119.03(19)	119.11(19)
N1–P2–S2	118.0(2)	116.7(2)	120.38(18)
P1–N1–P2	131.3(4)	131.9(3)	137.7(3)
S1–Te1–C5	86.1(2)	85.29(19)	86.0(2)
S1–Te1–C2	87.34(19)	83.33(18)	84.2(2)
S1–Te1···S2	78.49(5)	81.30(5)	96.96(4)
S1–Te1–S3	172.99(6)	169.47(5)	171.12(5)
S1–Te1···S4	124.27(5)	123.76(4)	125.78(5)
S2···Te1–C5	153.1(3)	79.76(19)	154.4(2)
S2···Te1–C2	73.64(19)	158.77(18)	121.53(18)
S2···Te1–S3	100.90(5)	102.60(5)	89.37(5)
S2···Te1···S4	123.37(6)	117.41(6)	71.63(4)
S3–C30–S4	119.9(4)	120.0(4)	120.8(4)
S3–Te1–C5	91.75(19)	85.79(19)	90.9(2)
S3–Te1–C2	85.79(19)	90.24(19)	87.2(2)
S3–Te1···S4	61.96(5)	63.37(5)	62.18(5)
S4···Te1–C5	83.6(3)	146.67(19)	86.0(2)
S4···Te1–C2	144.82(18)	83.40(18)	147.6(2)
Te1–S1–P1	89.36(7)	93.05(6)	97.98(6)
Te1–S2–P2	97.3(2)	93.93(8)	81.11(6)
Te1–S3–C30	97.5(3)	96.8(2)	96.6(2)
Te1–S4–C30	78.2(3)	79.7(2)	78.0(2)

Table 2
Torsion angles (°) for compounds $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$ (**1**), $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ (**2**) and $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$ (**4**)

	$[C_4H_8Te\{(SPPh_2)_2N\}_2]$ [a]		1	2	4
S2–Te1–S1–P1	−17.4	−29.5	−74.6	66.9	−14.2
Te1–S1–P1–N1	68.8	75.2	63.6	−51.8	63.4
Te1–S2–P2–N1	21.4	48.9	10.2	−12.6	67.2
S1–P1–N1–P2	−89.1	−52.0	25.5	−46.7	61.2
S2–P2–N1–P1	27.4	−25.7	−71.6	86.3	42.3
S1–Te1–S2–P2	−17.4	−16.0	48.0	−44.6	35.2

[a] Both TeS_2P_2N chelate rings display distorted folded geometry.

spectra were measured on a 3-nitrobenzyl-alcohol support in the positive ion mode on a JEOL JMS-SX102A instrument. The ^{1}H- (300 MHz), ^{13}C- (75 MHz), ^{31}P- (121 MHz) and ^{125}Te-NMR (94 MHz) spectra were recorded in $CDCl_3$ on a Jeol Eclipse + 300 spectrometer at room temperature using as external references 85% H_3PO_4 and $TeCl_4$. Elemental analyses were performed at Galbraith Laboratories (Knoxville, TN).

*3.2. $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$ (**1**)*

This was prepared by reaction of $C_4H_8TeI(S_2CNEt_2)$ (0.124 g, 0.24 mmol) with $[Na\{N(SPPh_2)_2\}]$ (0.195 g, 0.40 mmol) in ethanol (40 ml). The mixture was stirred at room temperature for 1 h. The resulting precipitate was filtered and dissolved in 15 ml of dichloromethane and filtered. The solution was then concentrated by pumping off 5 ml of the solvent, and adding then 10 ml of *n*-hexane. The resulting crystals are light yellow plates. Yield 0.123 g (57%), m.p. 145–146 °C. Anal. Found. C, 50.65; H, 4.91. Calc. for $C_{33}H_{38}N_2P_2S_4Te$ (780.48): C, 50.78; H, 4.91%. IR (KBr): 1483 ν(C–N), 1026/983 ν(C–S), 391 ν(S–Te), 1205, 912 ν(P=N), 696 ν(P–S), 567, 524 $\nu(C_2{-}Te)$ cm^{-1}. MS (FAB$^+$, CH_2Cl_2) *m/z*: 783 [M$^+$], 335 $[C_4H_8Te(S_2CNEt_2)^+]$, 186 $[C_4H_8Te^+]$, 448 $[(SPPh_2)_2N^+]$, 578 $[Te\{(SPPh_2)_2N\}^+]$. δ_H ($CDCl_3$) 1.19 (6H, t, CH_3), 3.76 (4H, q, $CH_2{-}CH_3$), 2.53 (4H, m, Te–CH_2–CH_2), 3.16 (4H, t, Te–CH_2), 7.33 (12H, P(S)–C_6H_5, m + p), 7.95 (8H, P(S)–C_6H_5, o); δ_C ($CDCl_3$) 12.3 (CH_3), 32.9 ($CH_2{-}CH_3$), 41.8 (Te–CH_2–CH_2), 48.8 (Te–CH_2), 128.1 (Cm, P(S)–C_6H_5, ^3J(PC) 12.7 Hz), 130.3 (Cp, P(S)–C_6H_5), 131.3 (Co, P(S)–C_6H_5), 138.6 (Ci, P(S)–C_6H_5), 195.2 (S_2CNR_2); δ_P ($CDCl_3$) 36.4; δ_{Te} ($CDCl_3$) 821.

*3.3. $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ (**2**)*

This was prepared following the procedure described for **1**, from $C_4H_8TeI(S_2CNC_5H_{10})$ (0.187 g, 0.40 mmol) and $[Na\{N(SPPh_2)_2\}]$ (0.281 g, 0.60 mmol). Yield 0.245 g (77%), m.p. 155–156 °C. Anal. Found: C, 51.87; H, 4.88. Calc. for $C_{34}H_{38}N_2P_2S_4Te$ (792.50): C, 51.53; H, 4.83%. IR (KBr): 1477 ν(C–N), 1023, 998 ν(C–S), 387 ν(S–Te), 1231, 913 ν(P=N), 696 ν(P–S), 570, 531 $\nu(C_2{-}Te)$ cm^{-1}. MS (FAB$^+$, CH_2Cl_2) *m/z*: 793 [M$^+$], 346 $[C_4H_8Te(S_2CNC_5H_{10})^+]$, 186 $[C_4H_8Te^+]$, 448 $[(SPPh_2)_2N^+]$, 578 $[Te\{(SPPh_2)_2N\}^+]$. δ_H ($CDCl_3$) 1.61 (6H, s, $(CH_2)_2(CH_2)$), 2.52 (4H, m, Te–CH_2–CH_2), 3.13 (4H, t, Te–CH_2), 3.97 (4H, s, N–CH_2), 7.32 (12H, P(S)–C_6H_5, m + p), 7.95 (8H, P(S)–C_6H_5, o); δ_C ($CDCl_3$) 23.7 (CH_2), 25.8 (N–CH_2–CH_2), 32.6 (N–CH_2), 33.4 (Te–CH_2–CH_2), 41.6 (Te–CH_2), 127.9 (Cm, P(S)–C_6H_5, ^3J(PC)14.7 Hz), 130.2 (Cp, P(S)–C_6H_5), 131.2 (Co, P(S)–C_6H_5, ^2J(PC) 10.32 Hz), 138.7 (Ci, P(S)–C_6H_5, ^1J(PC) 5.75 Hz), 195.1 (S_2CNR_2); δ_P ($CDCl_3$) 36.4; δ_{Te} ($CDCl_3$) 821.

*3.4. $[C_4H_8Te(S_2CNC_4H_8O)\{(SPPh_2)_2N\}]$ (**3**)*

This was prepared as described for **1**, from $C_4H_8TeI(S_2CNC_4H_8O)$ (0.188 g, 0.40 mmol) and $[Na\{N(SPh_2)_2\}]$ (0.281 g, 0.60 mmol). Yield 0.157 g (50%), m.p. 190 °C dec. Anal. Found: C, 49.89; H, 4.57. Calc. for $C_{33}H_{36}N_2OP_2S_4Te$ (794.47): C, 49.80; H, 4.53%. IR (KBr) 1470 ν(C–N), 1025, 991 ν(C–S), 386 ν(S–Te), 1213, 900 ν(P=N), 696 ν(P–S), 568, 517 $\nu(C_2{-}Te)$ cm^{-1}. MS (FAB$^+$, CH_2Cl_2) *m/z*: 795 [M$^+$], 634 $[C_4H_8Te\{(SPPh_2)_2N\}^+]$, 348 $[C_4H_8Te(S_2CNC_4H_8O)^+]$, 186 $[C_4H_8Te^+]$, 448 $[(SPPh_2)_2N^+]$, 578 $[Te\{(SPPh_2)_2N\}^+]$. δ_H ($CDCl_3$) 2.56 (4H, m, Te–CH_2–CH_2), 3.17 (4H, s, Te–CH_2), 3.69 (4H, t, (N–CH_2), 4.03 (4H, t, O–CH_2), 7.32 (12H, P(S)–C_6H_5, m + p), 7.91 (8H, P(S)–C_6H_5, o); δ_C ($CDCl_3$) 33.2 (Te–CH_2–CH_2), 42.4 (Te–CH_2), 50.96 (N–CH_2), 66.5 (O–CH_2), 128.1 (Cm, P(S)–C_6H_5, ^3J(PC) 12.0 Hz), 130.3 (Cp, P(S)–C_6H_5), 131.2 (Co, P(S)–C_6H_5, ^2J(PC) 15.0 Hz), 138.9 (Ci, P(S)–C_6H_5), 198.3 (S_2CNR_2); δ_P ($CDCl_3$) 36.4;); δ_{Te} ($CDCl_3$) 830.

*3.5. $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$ (**4**)*

This was prepared according to the procedure described for **1**, from $C_4H_8TeI(S_2CNC_4H_8S)$ (0.188 g, 0.40 mmol) and $[Na\{N(SPPh_2)_2\}]$ (0.281 g, 0.60 mmol). Yield 0.267 (82%), m.p. 152–153 °C. Anal. Found. C, 48.97; H 4.50. Calc. for $C_{33}H_{36}N_2P_2S_5Te$ (810.53): C,

Table 3
Crystallographic data and structure refinement parameters for the complexes $[C_4H_8Te(S_2CNEt_2)\{(SPPh_2)_2N\}]$ (**1**), $[C_4H_8Te(S_2CNC_5H_{10})\{(SPPh_2)_2N\}]$ (**2**) and $[C_4H_8Te(S_2CNC_4H_8S)\{(SPPh_2)_2N\}]$ (**4**)

	1	**2**	**4**
Empirical formula	$C_{33}H_{38}N_2P_2S_4Te$	$C_{34}H_{38}N_2P_2S_4Te$	$C_{33}H_{36}N_2P_2S_5Te$
Color	Yellow	Light-yellow	Yellow
Habit	Prism	Prism	Prism
Formula weight	780.43	792.44	810.48
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$C2/c$	$P2_1/c$	$P\bar{1}$
Unit cell dimensions			
a (Å)	35.123(4)	9.135(2)	9.243(1)
b (Å)	9.225(1)	17.418(3)	13.412(1)
c (Å)	22.125(3)	22.824(3)	16.221(1)
α (°)	90(1)	90(1)	110.86(1)
β (°)	105.40(1)	95.73(1)	97.25(1)
γ (°)	90(1)	90(1)	104.65(1)
Cell volume (Å^3)	6911.3(14)	3613.5(11)	1765.2(3)
Z	8	4	2
ρ_{calc} (g cm^{-3})	1.500	1.457	1.525
μ (mm^{-1})	1.221	1.169	1.256
Crystal size (mm)	0.68 × 0.30 × 0.20	0.68 × 0.44 × 0.12	0.60 × 0.34 × 0.14
2θ (°)	3.00–50.00	3.00–50.00	3.00–50.00
Reflections collected	6192	6278	5984
Independent reflections	6095 ($R_{int} = 0.0493$)	5881 ($R_{int} = 0.0356$)	5598 ($R_{int} = 0.0323$)
Absorption correction	Ψ-scans	Ψ-scans	Ψ-scans
Max./min. transmission	0.889/0.742	0.902/0.629	0.881/0.685
Parameters refined	399	389	388
Goodness-of-fit	1.024	1.030	1.038
Final R_1/wR_2	0.0489/0.1025	0.0442/0.0901	0.0435/0.0902

48.90; H 4.48%. IR (KBr) 1467 ν(C–N), 1026, 994 ν(C–S), 397 ν(S–Te), 1237, 916 ν(P=N), 696 ν(P–S), 557, 512 $\nu(C_2–Te)$ cm^{-1}. MS (FAB$^+$, CH_2Cl_2) m/z: 813 [M$^+$], 364 $[C_4H_8Te(S_2CNC_4H_8S)^+]$, 186 $[C_4H_8Te^+]$, 448 $[(SPPh_2)_2N^+]$, 578 $[Te\{(SPPh_2)_2N\}^+]$. δ_H ($CDCl_3$) 2.56 (4H, m, Te–CH_2–CH_2), 2.66 (4H, t, N–CH_2), 3.16 (4H, s, Te–CH_2), 4.31 (4H, t, S–CH_2), 7.31 (12H, P(S)–C_6H_5, m + p), 7.94 (8H, P(S)–C_6H_5, o); δ_C ($CDCl_3$) 27.6 (N–CH_2), 33.1 (Te–CH_2–CH_2), 42.4 (Te–CH_2), 54.3 (S–CH_2), 128.0 (Cm, P(S)–C_6H_5, ^3J(PC)13.8 Hz), 130.4 (Cp, P(S)–C_6H_5), 131.2 (Co, P(S)–C_6H_5), 138.7 (Ci, P(S)–C_6H_5), 197.9 (S_2CNR_2); δ_P ($CDCl_3$) 36.5; δ_{Te} ($CDCl_3$) 831.

3.6. Crystallographic studies

Suitable crystals for X-ray analysis of **1**, **2** and **4** were obtained by slow diffusion of n-hexane into dichloromethane solutions of the corresponding compound. None showed signals of decomposition during X-ray data collection, which was carried out at room temperature. Details of the data collection and refinement are summarized in Table 3. The structures were solved by direct methods using SHELXS-97 [22] and refined by full-matrix least-square calculations, using the program system SHELXL-97 [23]. Non-hydrogen atoms were refined anisotropically, whereas H atoms were refined as riding model with fixed isotropic U. In the case of **1**, there was disorder involving C3 and C4 atoms from the C_4H_8Te moiety. The best solution was obtained with occupancies of 0.57(3) for C3 and C4 and 0.43(3) for C3B and C4B.

4. Supplementary material

Crystallographic data for the structural analysis (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 160624 for **1**, 160625 for **2**, 160626 for **4**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: + 44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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