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Synthesis, spectroscopic characterization and structural studies of dialkyl dithiophosphinate and *N*,*N*-dialkyl dithio- and monothio-carbamate derivatives of 1-iodo-1,1,2,3,4,5-hexahydrotellurophene

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Abstract

The synthesis of the organotellurium(IV) compounds $[C_4H_8TeI(S_2PMe_2)]$ (**2**), $[C_4H_8TeI(S_2PEt_2)]$ (**3**), $[C_4H_8TeI(S_2CNC_4H_6)]$ (**4**) and $[C_4H_8TeI(SOCNC_5H_{10})]$ (**6**) was achieved. All of them were characterized by IR, ^1H-, ^{13}C-, ^{31}P- and ^{125}Te-NMR, mass spectroscopy, elemental analyses and single-crystal X-ray diffraction. In addition, the crystal and molecular structures of the previously known 1,1-diiodotetrahydrotellurophene and $[C_4H_8TeI(S_2CNEt_2)]$ (**5**) were obtained. The geometry around Te(IV) is that of a sawhorse structure in which the lone pair is apparently stereochemically active and occupying an equatorial position in a distorted trigonal bipyramid. All the structures exhibit different supramolecular associations. The intermolecular Te···S and Te···I interactions result in the formation of dimeric species in **4**, and in **1** and **6**, respectively. The intermolecular Te···S bonds in **2**, **3** and **5** lead to formation of one-dimensional polymers. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Dithiocarbamates; Dithiophosphinates; Monothiocarbamates; Structure; Tellurium(IV)

1. Introduction

During the last 30 years several papers have appeared on organotellurium(IV) compounds with 1,1-dithio ligands (see Ref. [1] for a review), including *N*,*N*-dialkyl dithiocarbamates [1–4], *O*-alkyl dithiocarbonates [4,5], dithiophosphates [3a,4,6], tetraphenyldithioimidophosphinates [7]. They exhibit a great structural diversity at tellurium geometry due to the strong tendency to form supramolecular associations through inter- and intramolecular Te···donor atom interactions and to the apparently stereochemically active lone pair at the tellurium center. The ligand bonding can usually be described as anisobidentate (unsymmetrical bidentate).

In contrast to the number of reported 1,1-dithiolato derivatives of the 1,3-dihydrobenzotellurophenes [4], few compounds containing the analogous tetrahydrotellurophene (**1**) have been well characterized. To date, only the syntheses of some derivatives have been reported: $C_4H_8TeI_{2-n}(dtc)_n$ (dtc = dimethyl-, diethyl-, piperidine- or morpholine-*N*-carbodithioate, and $n = 1$ or 2) [2b], and $C_4H_8Te(S_2COR)_2$ (R = Me, Et, $CHMe_2$) [8]. Recently, the crystal structure of some dicarboxylate derivatives was reported: $C_4H_8Te(OCOR)_2$ (R = C_6H_5, 4-$NO_2C_6H_4$ and $CCH{:}CHCO_2Bu$) [9].

In view of these features we considered it of interest to investigate a series of iododerivatives of the tetrahydrotellurophenes, C_4H_8TeIL, as intermediates to prepare mixed ligand compounds. We report here four new tellurium(IV) compounds: $[C_4H_8TeI(S_2PMe_2)]$ (**2**), $[C_4H_8TeI(S_2PEt_2)]$ (**3**), $[C_4H_8TeI(S_2CNC_4H_6)]$ (**4**) and $[C_4H_8TeI(SOCNC_5H_{10})]$ (**6**). They were characterized by IR, ^1H-, ^{13}C-, ^{31}P- and ^{125}Te-NMR, mass spectroscopy, elemental analyses and single-crystal X-ray diffraction. In addition, the crystal and molecular structures of the 1,1-diiodotetrahydrotellurophene (**1**) and $[C_4H_8TeI(S_2CNEt_2)]$ (**5**) are also reported.

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2. Results and discussion

The compounds reported were obtained in high yields by the reaction of $C_4H_8TeI_2$ (**1**) with equimolar amounts of the appropriate sodium salt of the ligand in CH_2Cl_2. The compounds are highly soluble in common organic solvents (CH_2Cl_2, $CHCl_3$, C_6H_6). They exhibit a gradual decomposition in both solution and solid state, giving tellurium and free ligand. This tendency to undergo reductive elimination has been observed in many of the known sulfur-bonding derivatives of organotellurium(IV) [2–6,10].

In the IR spectra of **2** and **3**, the difference between the asymmetric and symmetric $\nu(PS_2)$ vibration may be indicative of the co-ordination pattern of the ligand [11]. On this basis, the differences between the symmetric and asymmetric $\nu(PS_2)$ of 124 and 101 cm^{-1} for **2** and **3** are within the range for the monodentate co-ordination behavior. For compounds **4** and **5**, those containing 1,1-dithiothiocarbamates, the presence of two medium weak absorptions in the region 1040 to 970 cm^{-1} indicates anisobidentate behavior for both of the ligands [12]. A strong band at 1600 cm^{-1} $\nu(CO/CN)$ is consistent with monothiocarbamates groups being attached in monodentate mode [13].

The positive ion FAB mass spectra show low-intensity signals for the corresponding molecular ions, but rather intense fragments containing tellurium atom, i.e. $[C_4H_8Te]^+$ (186 m/z), $[C_4H_8TeI]^+$ (313 m/z), $[C_4H_8TeL]^+$ with $L = S_2CNC_4H_8$ (334 m/z), $S_2CNC_5H_6$ (330 m/z), S_2PMe_2 (311 m/z) and S_2PEt_2 (339 m/z). All of the observed signals exhibit the expected, characteristic, isotopic distribution patterns.

The ^1H- and ^{13}C-NMR spectra show the expected methylene protons and carbon atoms from the organoyltellurium moiety, as well as the methyl and methylene resonances from the ligands. The CH_2 directly attached to tellurium experiences an upfield shift relative to the starting material $C_4H_8TeI_2$. The resonances for the organic groups bonded to phosphorus in **2** and **3** exhibit the expected splitting pattern due to the phosphorus–proton and phosphorus–carbon couplings respectively. The ^{125}Te-NMR spectra also show phosphorous–tellurium coupling. The ^{31}P-NMR spectra of **2** and **3** and the ^{125}Te-NMR spectra of the compounds containing the dithiocarbamates (**4** and **5**) are seen as singlets. The ^{125}Te resonances of compounds **2** to **6** are shifted to high field compared with that of $C_4H_8TeI_2$ (782 ppm in $CDCl_3$). The partial substitution by the $[S_2CNR_2]$ group has more effect on the ^{125}Te chemical shift than that of the $[S_2PR_2]$ group.

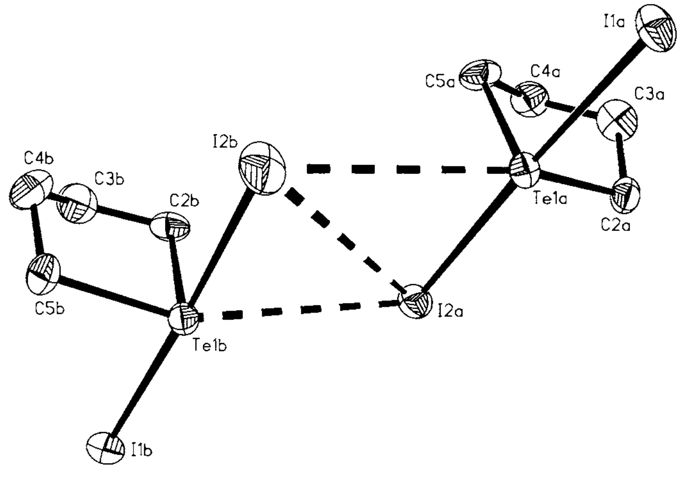

Fig. 1. ORTEP plot of the dimeric structure of **1**, $[C_4H_8TeI_2]_2$. Hydrogen atoms are omitted for clarity. The ellipsoids enclose 30% probability.

2.1. Description of the structures

The solid-state structure of compound **1** is shown in Fig. 1 with the atom numbering scheme. It consists of two crystallographically independent $C_4H_8TeI_2$ molecules that are associated through intermolecular Te···I interactions. The distance between the two iodine atoms (I2a and I2b) involved in these secondary interactions is within the sum of the van der Waals radii [14]. The structures of $[C_4H_8TeI(S_2PMe_2)]$ (**2**) and $[C_4H_8TeI(S_2PEt_2)]$ (**3**) are illustrated in Fig. 2, and those of $[C_4H_8TeI(S_2CNC_4H_6)]$ (**4**) and $[C_4H_8TeI(SOCN\text{-}C_5H_{10})]$ (**6**) are shown in Figs. 3 and 4 respectively. Selected bond lengths and angles, and crystallographic data for the $[C_4H_8TeI_2]_2$ and C_4H_8TeIL structures are listed in Tables 1 and 2 respectively.

In all cases the geometry around tellurium is best described as the sawhorse structure typical of tellurium(IV) compounds in which the lone pair is assumed to be stereochemically active and occupying an equatorial position in a distorted trigonal bipyramid [1–6,10]. The two carbon atoms occupy the other two equatorial positions with an average C–Te–C angle of 84.26° and average Te–C bond lengths (2.154 Å) close to the mean Te–C(alkyl) bond distance (2.158 Å) [15]. The axial positions are occupied by two iodine atoms in **1** and by the iodine atom and one of the sulfur atoms from the ligands in the C_4H_8TeIL derivatives (**2**–**6**). The I–Te–I and I–Te–S axial angles are within the range 176.04(4) to 176.59(5)° except for that of **4**, which is considerably smaller (170.89(7)°).

In the dimeric species of **1**, one iodine atom from each molecule interacts with an adjacent tellurium atom, to form two unsymmetrical Te–I···Te bridges with cis Te–I and Te···I links (see Fig. 1). Each Te···I bond is trans to one of the carbon atoms, C1a and C1b. The C–Te···I groups are non-linear with angles of 159.8(3) and 160.0(3)°.

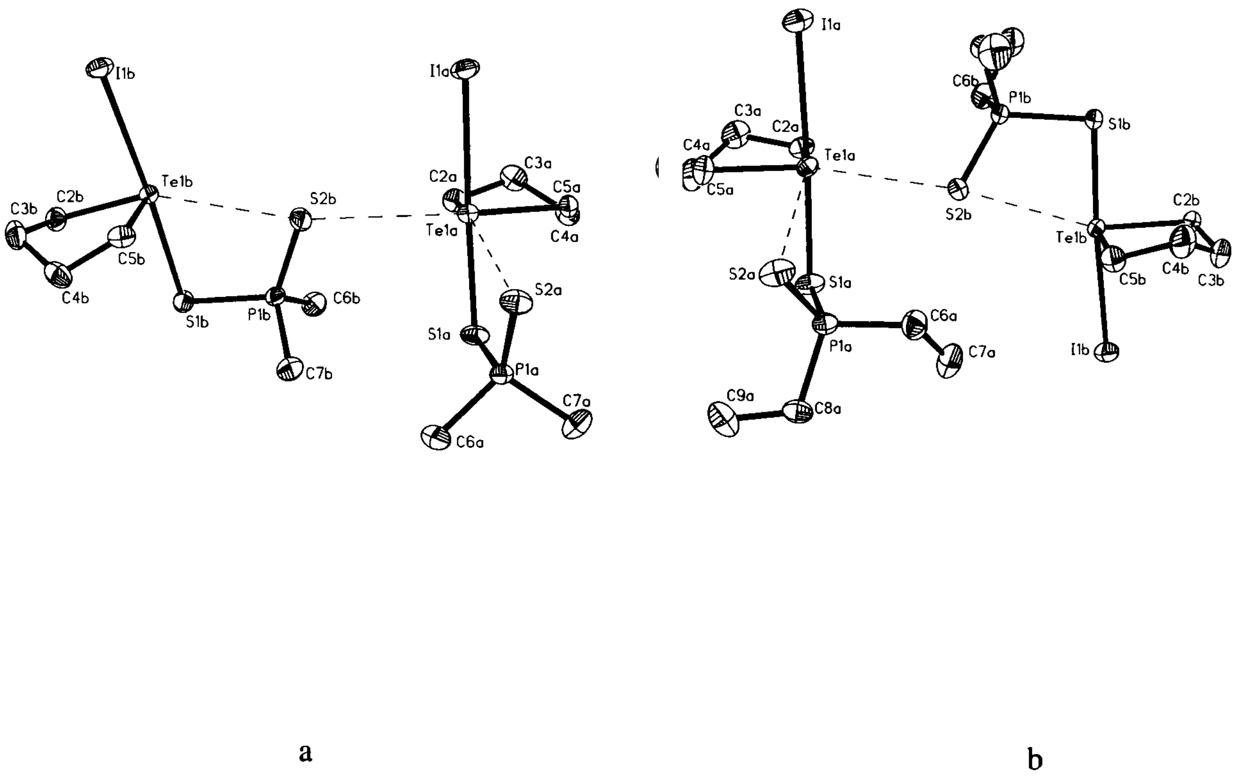

Fig. 2. ORTEP plot of (a) $[C_4H_8TeI(S_2PMe_2)]$ (**2**) and (b) $[C_4H_8TeI(S_2PEt_2)]$ (**3**). They show the Te···S interaction between two neighboring molecules. The ellipsoids enclose 30% probability.

In addition to the short I2a···I2b distance within the dimer, one of the iodine atoms from each associated molecule interacts (I···I bond of 4.067(2) Å) with the neighboring iodine atoms from the adjacent dimer, leading to a ladder structure.

The dithio ligands show anisobidentate behavior on interacting with the tellurium atom. The short Te–S bond distances in **2** to **5** cover the range 2.53 to 2.57 Å at an average of 2.556 Å, comparable with the reported range 2.484 to 2.58 Å for related R_2TeXL structures [1–4]. The long Te···S lengths range from 3.127(2) to 3.443(2) Å and are well within the sum of the van der Waals radii [14]. However, the two compounds containing the dithiophosphinate groups (**2** and **3**) exhibit the longest intramolecular Te···S distances and the two phosphorus–sulfur bonds are quite different (average 2.085 and 1.967 Å) and may be regarded as single (long) and double (short) P–S bonds. These and the observed $\Delta\nu(PS_2)_{s/as}$ in IR spectra suggest that the monodentate behavior is more suitable for these ligands [11]. The Te–I bond distances of the C_4H_8TeIL (**2** to **5**) are longer than those in $C_4H_8TeI_2$ (**1**) and related R_2TeI_2 species [16].

There are also close contacts between molecules in compounds **2** to **5**, but, unlike **1**, it is the aniso-bonded sulfur atom that forms the intermolecular Te···S bonds. In the case of **2**, **3** and **5**, this linking is extended to give a zigzag polymeric chain, whereas in **4** there are two molecules linked together in a dimer. In compound **2**, the phosphinate groups are cis along the polymeric chain, whereas in **3** the arrangement is trans with the largest I(1a)–Te(1a)···S(2b) and the smaller S(1a)–Te(1a)···S(2b) angles (see Fig. 2).

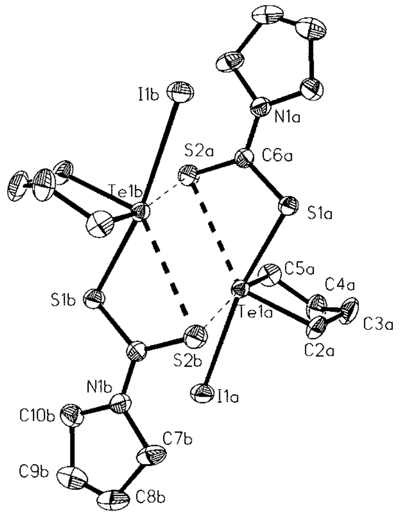

Fig. 3. ORTEP plot of two molecules of $[C_4H_8TeI(S_2CNC_4H_6)]$ (**4**) showing the dimeric association through intermolecular Te···S interaction The ellipsoids enclose 30% probability.

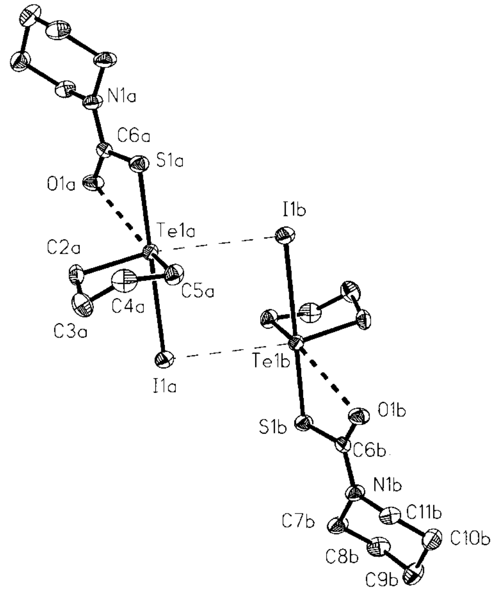

Fig. 4. ORTEP plot of two molecules of $[C_4H_8TeI(SOCNC_5H_{10})]$ (**6**) showing the dimeric association through intermolecular Te···I contacts. The ellipsoids enclose 30% probability.

In the compound containing the monothiocarbamate group (**6**), the Te–S bond lengths are shorter (2.490(2) Å) than those in **2** to **5**. Each oxygen atom is oriented toward a tellurium atom with a Te···O intramolecular distance of 2.844(6) Å. The C–O bond length of 1.223 Å is also comparable to those found in related R_2TeXL (L = monothiocarbamate) [10] and to those of terminal C=O bonds involved in secondary bonding, such as to a hydrogen atom [17]. The Te–I bond lengths are also longer than those in **2** to **5**. This lengthening of the Te–I bond and the shortening of the Te–S bonds were also observed in the related monothiocarbamate compounds and were claimed as examples of the trans effect operating on a main group metal [10]. The lengthening of the Te–I bond may also be due to the involvement of the iodine atom in an intermolecular Te···I bond, which leads to dimeric species like those observed in **1** and some other related haloderivatives [3,10].

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 methods described by Granger and coworkers [18]. The FAB mass spectra were measured on a 3-nitrobenzyl alcohol support in the positive ion mode on a JEOL JMS-SX102A instrument; the IR spectra (as KBr discs) were measured on a Nicolet FT-IR Magna 750 spectrometer. The ^1H- (300 MHz), ^{13}C- (75 MHz), ^{31}P- (121 MHz), ^{125}Te-NMR (94 MHz) spectra were recorded in $CDCl_3$ on a JEOL Eclipse + 300 spectrometer at room temperature using as external reference $Si(CH_3)_4$, H_3PO_4 85% and neat Me_2Te. Galbraith Laboratories (Knoxville, TN) performed microanalyses.

3.2. $[C_4H_8TeI(S_2PMe_2)]$ (**2**)

This was prepared by the reaction of $C_4H_8TeI_2$ (0.4 g, 0.9 mmol) with $Na(S_2PMe_2)$ (0.128 g, 0.9 mmol) in dichloromethane (50 ml). The solution was stirred overnight, filtered off, and evaporated to dryness. The crude products were stirred with *n*-hexane, filtered and washed with ethanol and water. Yield 0.143 g (71%), m.p. 98–99°C. Anal. Found: C, 16.33; H, 3.11. $C_6H_{14}IPS_2Te$ (435.76) requires C, 16.54; H, 3.24%. IR(KBr): 588s/464m $\nu(PS_2)_{asym/sym}$ cm^{-1}. MS (FAB$^+$, $CHCl_3$, for ^{130}Te) *m*/*z*: 438 $[M]^+$, 313 $[C_4H_8TeI]^+$, 311 $[C_4H_8TeIPS_2C_2H_6]^+$, 186 $[C_4H_8Te]^+$, 125 $[C_2H_6PS_2]^+$; δ_H ($CDCl_3$) 2.09 (6H, d, PCH_3, ^2J (PH) 12.9 Hz), 2.96 (4H, bs, $TeCH_2CH_2$), 3.9 (4H, bs, $TeCH_2$); δ_C ($CDCl_3$) 29.87 (d, PCH_3, ^1J (PC) 56.6 Hz), 34.85 ($TeCH_2\mathbf{C}H_2$), 44.3 ($Te\mathbf{C}H_2$); δ_P ($CDCl_3$) 55.62 (^1J (PC) 56.6 Hz); δ_{Te} ($CDCl_3$) 780.6 (^2J (PTe) 63 Hz).

3.3. $[C_4H_8TeI(S_2PEt_2)]$ (**3**)

This was prepared in a similar manner to **2** from $C_4H_8TeI_2$ (0.2 g, 0.45 mmol) and $Na(S_2PEt_2)]$ (0.079 g, 0.45 mmol). Yield 0.110 g (52%), m.p. 134°C. Anal. Found: C, 20.46; H, 3.74. $C_8H_{18}IPS_2Te$ (463.81) requires C, 20.72; H, 3.91%. IR(KBr): 586s/485m $\nu(PS_2)_{asym/sym}$ cm^{-1}. MS (FAB$^+$, $CHCl_3$, for ^{130}Te) *m*/*z*: 339 $[C_4H_8TeIPS_2C_4H_{10}]^+$, 313 $[C_4H_8TeI]^+$, 186 $[C_4H_8Te]^+$, 153 $[C_4H_{10}PS_2]^+$; δ_H ($CDCl_3$) 1.26 (6H, dt, $PCH_2\mathbf{CH_3}$, ^3J (PH) 21.7 Hz), 2.09 (6H, dq, $P\mathbf{CH_2}$, ^2J (PH) 10.4 Hz), 2.83 (4H, m, $TeCH_2\mathbf{CH_2}$), 3.74 (4H, m, $Te\mathbf{CH_2}$); δ_C ($CDCl_3$) 7.09 (d, $PCH_2\mathbf{C}H_3$, ^2J (PC) 4.6 Hz), 31.8 (d, $P\mathbf{C}H_2$, ^1J (PC) 52 Hz), 34.88 ($TeCH_2\mathbf{C}H_2$), 44.25 ($Te\mathbf{C}H_2$); δ_P ($CDCl_3$) 76.89 (^1J (PC) 52 Hz); δ_{Te} ($CDCl_3$) 779.66 (^2J (PTe) 57 Hz).

3.4. $[C_4H_8TeI(S_2CNC_4H_6)]$ (**4**)

This was prepared in a similar manner to **2** from $C_4H_8TeI_2$ (0.4 g, 0.9 mmol) and $Na(S_2CNC_4H_6)$ (0.15 g, 0.9 mmol).Yield 0.250 g (84%), m.p.(dec.) 146–150°C. Anal. Found: C, 23.42; H, 2.97. $C_9H_{14}INS_2Te$ (454.83) requires C, 23.77; H, 3.10%. IR(KBr): 1431s ν(CN), 1040mw/991m $\nu(CS_2)$ cm^{-1}. MS (FAB$^+$, $CHCl_3$, for ^{130}Te) *m*/*z*: 475 $[M]^+$, 330 $[C_4H_8TeS_2NC_5H_6]^+$, 313 $[C_4H_8TeI]^+$, 186 $[C_4H_8Te]^+$, 144 $[C_5H_6NS_2]^+$; δ_H ($CDCl_3$) 2.78 (4H, bs, $TeCH_2\mathbf{CH_2}$), 3.4 (4H, bs, $Te\mathbf{CH_2}$), 4.45 (4H, s, $N\mathbf{CH_2}$), 5.87 (2H, s, $NCH_2\mathbf{CH}$);

δ_C (CDCl$_3$) 33.37 ($\mathrm{TeCH_2\mathbf{C}H_2}$), 41.4 ($\mathrm{Te\mathbf{C}H_2}$), 59.67 ($\mathrm{N\mathbf{C}H_2}$), 125.66 ($\mathrm{NCH_2\mathbf{C}H}$), 192.3 ($\mathrm{S_2\mathbf{C}N}$); δ_{Te} (CDCl$_3$) 775.5.

3.5. [$C_4H_8TeI(S_2CNEt_2)$] (**5**)

This was prepared in a similar manner to **2** from $C_4H_8TeI_2$ (0.2 g, 0.45 mmol) and Na(S_2CNEt_2) (0.077 g, 0.45 mmol).Yield 0.172 g (82%), m.p. 138–141°C (123°C (dec.) [2b]) Anal. Found: C, 23.38 (C, 21.05 [2b]); H, 3.85 (H, 3.92 [2b]). $C_9H_{18}INS_2Te$ (458.86) requires C, 23.56; H, 3.95%. IR (KBr): 1482s (1498 [2b]) ν(CN), 1025mw/980m (988m/972vs [2b]) $\nu(CS_2)$ cm^{-1}. MS (FAB$^+$, CHCl$_3$, for ^{130}Te) m/z: 461 [M]$^+$, 334 [$C_4H_8TeS_2NC_4H_{10}$]$^+$, 313 [C_4H_8TeI]$^+$, 186 [C_4H_8Te]$^+$, 148 [$C_4H_8NS_2$]$^+$; δ_H (CDCl$_3$) 1.25 (6H, t, $\mathrm{NCH_2C\mathbf{H}_3}$), 2.7 (4H, bs, $\mathrm{TeCH_2C\mathbf{H}_2}$), 3.33 (4H, bs, $\mathrm{TeC\mathbf{H}_2}$), 3.67 (4H, q, $\mathrm{NC\mathbf{H}_2CH_3}$); δ_C (CDCl$_3$) 12.28 ($\mathrm{NCH_2\mathbf{C}H_3}$), 32.95 ($\mathrm{TeCH_2\mathbf{C}H_2}$), 40.55 ($\mathrm{Te\mathbf{C}H_2}$), 48.45 ($\mathrm{N\mathbf{C}H_2CH_3}$), 194.2 ($\mathrm{S_2\mathbf{C}N}$); δ_{Te} (CDCl$_3$) 772.52.

3.6. [$C_4H_8TeI(SOCNC_5H_{10})$] (**6**)

This was prepared in a similar manner to **2** from $C_4H_8TeI_2$ (0.4 g, 0.9 mmol) and Na($SOCNC_5H_{10}$) (0.26 g, 0.9 mmol).Yield 0.34 g (84.1%), m.p.(dec.) 130°C. Anal. Found: C, 26.32; H, 3.90. $C_9H_{14}INOSTe$ (454.82) requires C, 26.41; H, 3.99%. IR(KBr): 1600vs and 1405s ν(CO/CN). MS (FAB$^+$, CHCl$_3$, for ^{130}Te) m/z: 456 [M]$^+$, 330 [$C_4H_8TeSONC_5H_6$]$^+$, 313 [C_4H_8TeI]$^+$, 186 [C_4H_8Te]$^+$, 112 [$C_6H_{10}NO$]$^+$; δ_H (CDCl$_3$) 2.68 (4H, bs, $\mathrm{TeCH_2C\mathbf{H}_2}$), 3.34 (4H, bs, $\mathrm{TeC\mathbf{H}_2}$), 3.48 (4H, bs, $\mathrm{NC\mathbf{H}_2}$), 1.57 (6H, bs, $\mathrm{NCH_2(C\mathbf{H}_2)_3}$); δ_C (CDCl$_3$) 33.84 ($\mathrm{TeCH_2\mathbf{C}H_2}$), 41.54

Table 1
Selected bond lengths (Å) and angles (°) for the two independent [$C_4H_8TeI_2$] molecules (a and b) of compound **1**, and [C_4H_8TeIL] (L = S_2PMe_2 (**2**), S_2PEt_2 (**3**), $S_2CNC_4H_6$ (**4**), S_2CNEt_2 (**5**) and $SOCNC_5H_{10}$ (**6**))

	1		**2**	**3**	**4**	**5**	**6**
	molecule a	molecule b					
Te(1a)–C(2a)	2.167(12)	2.160(12)	2.143(7)	2.153(6)	2.163(10)	2.148(7)	2.149(7)
Te(1a)–C(5a)	2.181(12)	2.175(12)	2.147(7)	2.155(7)	2.136(10)	2.150(9)	2.152(8)
Te(1a)–I(1a)	2.891(2)	2.966(2)	2.9599(8)	3.029(1)	3.046(1)	3.0395(9)	3.1203(7)
Te(1a)–I(2a)	2.993(2)	2.914(2)					
Te(1a)···I(2b)	4.019(2)	3.813(2)					
Te(1a)–S(1a)			2.578(2)	2.564(2)	2.550(3)	2.534(2)	2.490(2)
N(11a)–C(6a)					1.334(13)	1.323(10)	1.351(10)
Te(1a)···I(1b)							3.7031(7)
Te(1a)···O(1a)							2.844(6)
Te(1a)···S(2b)			3.302(2)	3.443(2)	3.266(3)	3.127(2)	
Te(1a)···S(2b)			3.553(2)	3.613(2)	3.603(3)	3.582(2)	
I(1a)–Te(1a)–I(2a)	176.5(1)	177.6(1)					
C(2a)–Te(1a)–C(5a)	84.0(5)	84.6(5)	83.8(3)	84.8(3)	84.6(5)	84.1(3)	84.6(3)
C(2a)–Te(1a)–I(1a)	88.4(3)	88.3(4)	89.1(2)	88.08(17)	87.2(3)	87.0(2)	80.2(3)
C(5a)–Te(1a)–I(1a)	92.1(4)	89.3(3)	89.3(2)	88.6(2)	87.7(3)	90.4(3)	88.7(3)
C(2a)–Te(1a)–I(2a)	88.1(3)	91.1(4)					
C(5a)–Te(1a)–I(2a)	88.0(4)	88.3(3)					
C(2a)–Te(1a)···I(2b)	160.6(4)	80.5(4)					
C(5a)–Te(1a)···I(2b)	90.5(4)	158.9(4)					77.2(2)
C(2a)–Te(1a)···I(1b)							161.2(2)
C(2a)–Te(1a)–S(1a)			88.2(2)	88.65(18)	83.7(3)	92.6(3)	92.6(2)
C(5a)–Te(1a)–S(1a)			88.1(2)	88.8(2)	91.2(3)	86.1(3)	87.8(3)
I(1a)–Te(1a)–S(1a)			176.59(5)	176.04(4)	170.89(7)	176.50(5)	176.37(5)
I(1a)–Te(1a)···S(2a)			113.1(5)	115.31(4)	127.0(3)	120.39(4)	
I(1a)–Te(1a)···O(1a)							124.36(11)
I(1a)–Te(1a)···S(2b)			91.1(5)	104.87(4)	86.9(3)	92.96(5)	
S(1a)–Te(1a)···S(2b)			90.6(3)	76.3(2)	93.2(3)	85.66(6)	
S(1a)–Te(1a)···S(2a)			69.0(3)	67.3(2)	61.6(3)	62.95(6)	
S(1a)–Te(1a)···O(1a)							58.71(12)
S(2a)···Te(1a)···S(2b)			109.0(3)	111.3(2)	107.7(4)	122.74(7)	
I(1a)–Te(1a)···I(1b)							88.774(18)
Te(1a)–I(2a)···Te(1b)	106.3(3)	103.0(3)					
Te(1a)–S(1a)–P(1a)/C(6a)			97.50(8)	94.83(8)	98.7(3)	97.3(3)	90.7(2)
Te(1a)–O(1a)–C(6a)							89.6(4)
S(1a)–P(1a)/C(6a)–S(2a)			114.7(1)	113.7(1)	123.0(6)	120.4(5)	
S(1a)–C(6a)–O(1a)							120.3(6)

Table 2
Crystallographic data for compound **1**, and [C_4H_8TeIL] (L = S_2PMe_2 (**2**), S_2PEt_2 (**3**), $S_2CNC_4H_6$ (**4**), S_2CNEt_2 (**5**) and $SOCNC_5H_{10}$ (**6**))

	1	**2**	**3**	**4**	**5**	**6**
Formula	$C_8H_{16}I_4Te_2$	$C_6H_{14}IPS_2Te$	$C_8H_{18}IPS_2Te$	$C_9H_{14}INS_2Te$	$C_9H_{18}INS_2Te$	$C_{10}H_{18}INOSTe$
Color; habit	Red-wine; prism	Yellow; block	Yellow; prism	Yellow; prism	Yellow; prism	Yellow; prism
Formula mass	875.011	435.76	463.81	454.83	458.86	454.81
Cryst. size (mm)	0.38 × 0.16 × 0.16	0.50 × 0.40 × 0.32	0.60 × 0.52 × 0.24	0.40 × 0.40 × 0.24	0.44 × 0.38 × 0.20	0.76 × 0.36 × 0.14
Diffractometer	Siemens P4/PC	Siemens P4/PC	Siemens P4/PC	Siemens P4/PC	Siemens P4/PC	Siemens P4/PC
λ (Mo–K_α) (Å)	0.710 73	0.710 73	0.710 73	0.710 73	0.710 73	0.710 73
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$ (no. 14)	$P2_1/n$ (no. 14)	$P2_1/c$ (no. 14)	$P2_1/c$ (no. 14)	$P2_1/c$ (no. 14)	$P2_1$/n (no. 14)
a (Å)	12.736(5)	10.666(2)	10.070(3)	6.491(1)	10.818(2)	6.294(1)
b (Å)	10.426(4)	12.519(2)	11.607(3)	19.752(6)	11.690(1)	20.424(1)
c (Å)	13.948(5)	10.934(2)	13.788(4)	11.202(3)	11.763(1)	11.547(1)
α (°)	90	90	90	90	90	90
β (°)	90.78(1)	117.50(1)	108.62	103.25(1)	92.75(1)	102.86(1)
γ (°)	90	90	90	90	90	90
Z	4	4	4	4	4	4
μ (mm^{-1})	9.794	5.078	4.313	4.603	4.331	4.312
Absorption correction	ψ scan	ψ scan	ψ scan	ψ scan	ψ-scan	ψ-scan
2θ range (°)	3.0 to 50.0	3.0 to 50.0	3.0 to 50.0	3.0 to 50.0	3.0 to 50.0	3.0 to 50.0
Reflections collected	4190	2403	2845	2535	2575	2611
Independent reflections (R_{int})	3269 (0.0375)	2277 (0.0256)	2685 (0.0279)	23 114 (0.0308)	2440 (0.0248)	2383 (0.0394)
R (F_o)[a]	0.0409	0.0349	0.0353	0.0477	0.0384	0.0374
R_w (F_o)[b]	0.0884	0.0822	0.0768	0.1152	0.0841	0.0959
Goodness-of-fit	1.064	1.104	1.073	1.070	1.016	1.092

[a] $R_1 = \Sigma\|F_o|-|F_c\|/\Sigma|F_o|$.

[b] $wR_2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

(Te**C**H_2), 49.7 (N**C**H_2), 24.21 (NCH_2(**C**H_2)$_3$), 167.3 (S_2**C**N); δ_{Te} ($CDCl_3$) 772.46.

3.7. Crystallography

Crystals suitable for X-ray analysis were obtained by slow diffusion of *n*-hexane into dichloromethane solutions of the corresponding compound. Details of the data collections and refinements are summarized in Table 2. The structures were solved by direct methods and refined by full-matrix least-squares calculations, using the program system SHELXL-97 [19]. Non-hydrogen atoms were refined anisotropically, whereas H atoms were refined using a riding model.

4. Supplementary material

Crystallographic data for the structural analysis (excluding structure factors) has been deposited with the Cambridge Crystallographic Data Centre, CCDC 144864 (**1**), CCDC 144865 (**2**), CCDC 144866 (**3**), CCDC 144867 (**4**), CCDC 144868 (**5**) and CCDC 144869 (**6**). 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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