

# Synthesis, spectroscopic characterization of *O,O*-alkylene dithiophosphates of tellurolane and 1-oxa-4-tellurane. Single crystal structures of $C_4H_8Te[S_2P(OCH_2)_2CMe-{}^nPr]_2$ and $C_4H_8OTe[S_2P(OCH_2)_2CEt_2]_2$

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Received 16 December 2004; revised 27 January 2005; accepted 28 January 2005

Available online 11 May 2005

## Abstract

The synthesis of the four disubstituted organotellurium (IV) compounds,  $C_4H_8Te[S_2P(OCH_2)_2CEt_2]_2$  (**1**),  $C_4H_8Te[S_2P(OCH_2)_2CMe-{}^nPr]_2$  (**2**),  $C_4H_8OTe[S_2P(OCH_2)_2CEt_2]_2$  (**3**),  $C_4H_8OTe[S_2P(OCH_2)_2CMe-{}^nPr]_2$  (**4**), and one monosubstituted compound  $C_4H_8OTe(D)[S_2P(OCH_2)_2CMe-{}^nPr]$  (**5**), was achieved. All compounds were characterized by IR, EI, MS, and multinuclear NMR ( ${}^1H$ ,  ${}^{13}C$ ,  ${}^{31}P$ ,  ${}^{125}Te$ ). In addition, the molecular structures of **2** and **3** were determined by X-ray analyses. Both structures are monomeric and the tellurium atom is bonded to two carbon atoms and to one sulfur atom of each one of the two anisobidentate dithiophosphate ligands. In **2**, the geometry around Te(IV) is as that of a sawhorse structure in which the lone pair is apparently stereochemically active and occupying an equatorial position in a  $\psi$ -trigonal bipyramid. However, when the second sulfur atom of the dithiophosphate ligand is included in the coordination sphere, the environment about tellurium in the compound **2** can be described as bicapped  $\psi$ -trigonal bipyramid. Whereas in **3** it is a distorted octahedral structure in which the lone pair is stereochemically inactive.

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**Keywords:** Alkylene-dithiophosphate; Tellurium; Organotellurium (IV); Tellurolane; Tellurane; Crystal structure

## 1. Introduction

Several reports on organotellurium (IV) compounds with 1,1-dithioligands are known, including *O*-alkyl-dithiocarbonate [1–3], *N,N*-dialkyl-dithiocarbamates [4–7], *O,O*-dialkyl and alkylene dithiophosphates [8–15]. They exhibit a great structural diversity due mainly to the strong inclination to form

supramolecular associations through inter- and intramolecular Te–donor atom interactions and to the apparently stereochemically active lone pair at the tellurium center. Furthermore, these compounds show a greater tendency to undergo reductive-elimination to tellurium (II) and to the corresponding disulfide [14].

Only few derivatives of the type  $R_2Te[S_2PO_2G]_2$  [14–18], where R = Me, Ph, *p*-MeOPh,  $C_8H_8$ , and the cyclic dithiophosphate are of five or six-membered with different substitution pattern ( $G = -CMe_2CMe_2-$ ,  $-CHMeCHMe-$ ,  $-CH_2CMe_2CH_2-$ ,  $-CH_2CEt_2CH_2-$ ,

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–CHMeCH<sub>2</sub>CMe<sub>2</sub>–), are known, while reports with hetero-tellurium (IV) have received much less attention, despite the fact that they are less susceptible to reductive elimination to tellurium (II) than the linear ones [19,20].

We considered of interest to investigate the products of the reaction between two heterocyclic tellurium (IV) compounds: 1,1-diiodotellurolane, C<sub>4</sub>H<sub>8</sub>TeI<sub>2</sub> and 1-oxa-4,4-diiodotellurane, C<sub>4</sub>H<sub>8</sub>OTeI<sub>2</sub> with the sodium salt of two cyclic dithiophosphates, Na[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt<sub>2</sub>] and Na[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr]. We report here five new tellurium (IV) compounds, where four are disubstituted C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt<sub>2</sub>]<sub>2</sub> (**1**), C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr]<sub>2</sub> (**2**), C<sub>4</sub>H<sub>8</sub>OTe[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt<sub>2</sub>]<sub>2</sub> (**3**), C<sub>4</sub>H<sub>8</sub>OTe[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr]<sub>2</sub> (**4**) and one is monosubstituted C<sub>4</sub>H<sub>8</sub>OTe[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr] (**5**). They were characterized by mass spectrometry, IR, multinuclear NMR (<sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P and <sup>125</sup>Te). The molecular structures of two compounds **2** and **3** determined for single-crystal X-ray diffraction are also reported.

## 2. Results and discussion

The reaction between 1,1-diiodotellurolane or 1-oxa-4-iodotellurane with the sodium salts of 5',5'-diethyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate and 5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate, in chloroform or ethanol gave the corresponding disubstituted derivatives: tellurolane-1,1-diyl bis(5',5'-diethyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate) (**1**), tellurolane-1,1-diyl bis(5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate) (**2**), 1-oxa-4-tellurane-4,4-diyl bis(5',5'-diethyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate) (**3**), 1-oxa-4-tellurane-4,4-diyl bis(5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate) (**4**) and one monosubstituted 1-oxa-4-tellurane-4-iodide-4-(5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate) (**5**). The obtained compounds are solids and varying from white to pale yellow, soluble in common organic solvents such as CHCl<sub>3</sub>, CH<sub>2</sub>Cl<sub>2</sub>, Me<sub>2</sub>CO, benzene, etc. They are stable towards atmospheric oxygen and moisture, however, they decompose slowly in both solution and solid state.

At first, we tried to obtain monosubstituted and also disubstituted derivatives, however, only one monosubstituted compound was formed, concluding that dithiophosphate ligands show an important tendency to disubstitution.

The compounds were characterized by IR, electron-impact (EI) mass spectrometry and multinuclear NMR (<sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P and <sup>125</sup>Te) spectroscopy. The crystal and molecular structures of **2** and **3** were determined by single-crystal X-ray techniques.

IR spectra of these new compounds have been examined in KBr as discs in the region 4000–500 cm<sup>-1</sup>, and tentative assignments have been made on the basis of earlier reports [21,22] for similar compounds. The bands present in the regions 1090–970 and 820–800 cm<sup>-1</sup> are assigned to ν(P–O–C) and ν(P–O–(C)). A strong to medium band in the region 950–910 cm<sup>-1</sup> is due to dithiophosphate rings. A strong band present in the region 675–670 cm<sup>-1</sup> is assigned to ν(P=S). The bands of medium intensity in the region 520–500 cm<sup>-1</sup> may be attributed to ν(P–S) vibrations.

The mass spectra of the obtained Te (IV) compounds **1**–**5** were recorded with EI<sup>+</sup> technique. Only the compound **4** shows in its spectra a low intensity signal corresponding to the molecular ion (M<sup>+</sup>, 652 *m/z*). Besides, fragments containing tellurium atom with ligand parts or fragments derivatives of the ligands, i.e., [C<sub>4</sub>H<sub>8</sub>S<sub>4</sub>P<sub>2</sub>Te]<sup>+</sup> (376 *m/z*), [C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>PS<sub>2</sub>Te]<sup>+</sup> (352 *m/z*), [C<sub>4</sub>H<sub>8</sub>S<sub>2</sub>PTe]<sup>+</sup> (281 *m/z*), for compound **1**, [C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>SPTe]<sup>+</sup> (322 *m/z*), [C<sub>4</sub>H<sub>6</sub>O<sub>2</sub>S<sub>2</sub>P]<sup>+</sup> (181 *m/z*), [C<sub>4</sub>H<sub>6</sub>O<sub>2</sub>SP]<sup>+</sup> (150 *m/z*), for compound **2**, [C<sub>14</sub>H<sub>28</sub>O<sub>4</sub>S<sub>4</sub>P<sub>2</sub>]<sup>+</sup> (450 *m/z*) for compound **3**, [C<sub>11</sub>H<sub>22</sub>O<sub>3</sub>S<sub>2</sub>PTe]<sup>+</sup> (427 *m/z*), C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>PS<sub>2</sub>]<sup>+</sup> (225 *m/z*) for compound **4**, [C<sub>14</sub>H<sub>28</sub>O<sub>4</sub>S<sub>4</sub>P<sub>2</sub>]<sup>+</sup> (450 *m/z*), [C<sub>11</sub>H<sub>22</sub>O<sub>3</sub>S<sub>2</sub>PTe]<sup>+</sup> (427 *m/z*), [C<sub>8</sub>H<sub>15</sub>O<sub>3</sub>S<sub>2</sub>PTe]<sup>+</sup> (384 *m/z*), [C<sub>4</sub>H<sub>8</sub>OTe]<sup>+</sup> (329 *m/z*), [I<sub>2</sub>]<sup>+</sup> (254 *m/z*) for compound **5**, are observed; the three last compounds present the fragment, [C<sub>4</sub>H<sub>8</sub>OTe]<sup>+</sup> (202 *m/z*). All of the observed signals exhibit the expected characteristic isotopic distribution patterns.

The <sup>1</sup>H NMR spectral data show the characteristic proton resonances of corresponding cyclic dithiophosphate as well as 1,1-diiodotellurolane and 1-oxa-4,4-iodotellurane. The splitting pattern and intensities of peaks in the spectra of all these compounds are found to be consistent with its structures. The methylene directly attached to tellurium experiences an upfield shift relative to the starting materials C<sub>4</sub>H<sub>8</sub>TeI<sub>2</sub> and C<sub>4</sub>H<sub>8</sub>OTeI<sub>2</sub>.

The values of the chemical shifts within the dithiophosphate groups are found to be similar to those of the salts from which they were prepared so that there are no significant changes as a result of being linked to tellurium. The methylene protons of the 1,3,2-dioxaphosphinane ring show coupling with phosphorus to give doublets in the case of **1** and **3** and double of double signals for compounds **2**, **4** and **5**; the presence of two signals in these last compounds is due to the diastereotopic situation of the methylene protons. The NMR signals of these methylene protons are indicative of a rapid interchange between the two sulfur atoms similar to that reported for other complexes [15]. Furthermore, presenting very similar values for *J*<sub>(PCCH)</sub> in the range 15.60–16.00 Hz, corresponding to the average value for the sum of coupling constants <sup>3</sup>*J*<sub>P-H<sub>cis</sub></sub> and <sup>3</sup>*J*<sub>P-H<sub>trans</sub></sub> for a chair conformation situation, suggests that these compounds present a conformational equilibrium in solution, with equally populated chair conformers.

The  $^{13}\text{C}$  NMR spectral data show the expected carbon atoms and that the two dithiophosphate ligands are equivalent for disubstituted derivatives here reported. The carbon atoms of the tellurium ring experience an upfield shift relative to the starting materials  $\text{C}_4\text{H}_8\text{TeI}_2$  and  $\text{C}_4\text{H}_8\text{OTeI}_2$ . The signals attributable to all of the carbon atoms of the cyclic dithiophosphate groups are consistent with its structures and show splitting due to the phosphorus–carbon couplings. The doublets arising from P–C coupling and the values of the  $J_{\text{P-C}}$  coupling constants are similar to those observed for the bis compounds and related cyclic dithiophosphate derivatives [15].

The  $^{31}\text{P}$  NMR spectral data show the presence of a single peak in all compounds, indicative of the presence of only one species and also that the two dithiophosphate groups are in identical environments. It has been proposed that the difference in the phosphorus chemical shift between the corresponding free acid and the complex is a strong indicator of the mode of binding [23]. Downfield shifts in the range 8–15 ppm were claimed to be consistent with the presence of bidentate ligands, while negligible shifts or even upfield shifts were associated with monodentate linkages. In our compounds, the downfield shifts range from 14.07 to 18.40 ppm, which is consistent with the presence of bidentate ligands in solution rather than anisobidentate as found in the solid state structures and also indicative of a rapid interchange between the two sulfur atoms, in a similar process found in phosphorus compounds containing five- and six-membered ring [15,17].

$^{125}\text{Te}$  NMR shows that only a single peak was observed for five compounds. The resonances compounds are shifted slightly to downfield compared with that of  $\text{C}_4\text{H}_8\text{TeI}_2$  (796 ppm,  $\text{CDCl}_3$ ) and  $\text{C}_4\text{H}_8\text{OTeI}_2$  (555.9 ppm,  $\text{CDCl}_3$ ). The data fall in the range of 851.72–851.87 ppm for compounds **1** and **2**, and 557.00–565.89 pm for compounds **3–5** and the shift for disubstituted derivative **4** is downfield than that found in the monosubstituted derivative **5**.

### 3. Description of the structures

The solid-state structures of compounds  $\text{C}_4\text{H}_8\text{Te}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe-}^n\text{Pr}]_2$  **2** and  $\text{C}_4\text{H}_8\text{OTe}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CEt}_2]_2$  **3** are shown in Figs. 1 and 2 with the atom numbering Scheme 1. Crystallographic data and selected bond lengths and angles for the structures are listed in Tables 1 and 2, respectively. These compounds crystallize in discrete molecular units, where the geometry about tellurium can be described as being based on a distorted saw horse structure or a  $\psi$ -trigonal bipyramid in which a long pair is assumed to be active stereochemically and occupies a basal position. This type of geometry has been observed previously in a variety of acyclic and cyclic tellurium compounds [2,4–6,9,15,17,19,20]. The two adjacent carbon atoms occupy the other two equatorial positions with C–Te–C bond angles of  $84.8(4)^\circ$  and  $93.0(3)^\circ$  and average Te–C bond lengths 2.133 and 2.154 Å in **2** and **3**, being close to the value of  $(84.5(4)^\circ, 2.152 \text{ \AA})$  in  $\text{C}_4\text{H}_8\text{TeI}_2$  [24] and  $(94.1(4)^\circ,$

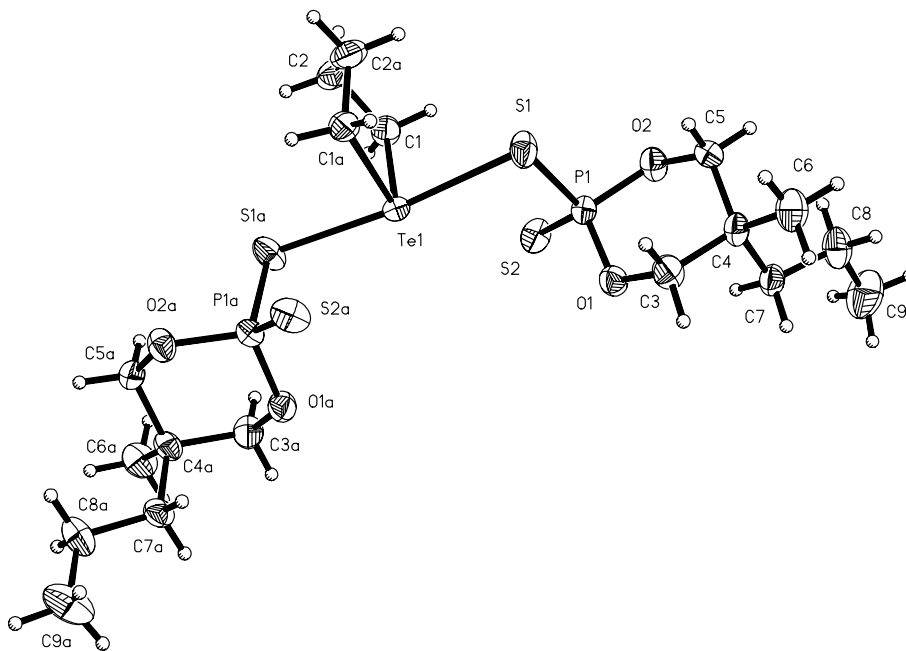


Fig. 1. ORTEP plot of the structure of **2**,  $\text{C}_4\text{H}_8\text{Te}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe-}^n\text{Pr}]_2$ . Hydrogen atoms are omitted for clarity. The ellipsoids enclose 30% probability.

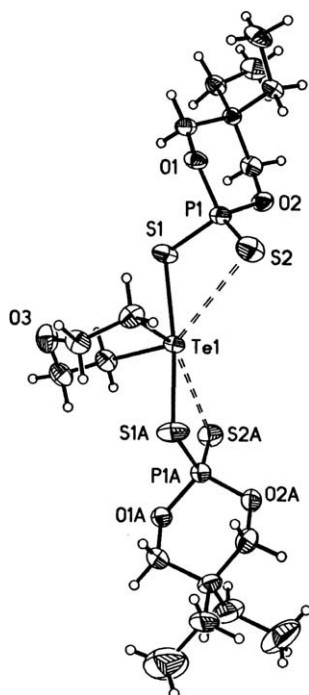


Fig. 2. ORTEP plot of the structure of **3**,  $C_4H_8OTe[S_2P(OCH_2)_2CET_2]_2$ .

2.16 Å) in  $C_4H_8OTeI_2$  [26].  $[C_4H_8TeI(S_2PMe_2)]$  and  $[C_4H_8TeI(S_2PET_2)]$  are also similar to those reported for the related species, which are in the range from  $83.8(3)^\circ$  to  $84.8(3)^\circ$  for bond angles and from 2.143 to 2.155 Å for the bond lengths [25,26].

The dithiophosphate ligands show an anisobidentate behavior on interacting with the tellurium atom. The short Te–S bonds in the apical positions are of Te(1)–S(1) 2.6410(18) Å, Te(1)–S(1a) 2.6410(18) Å for **2** and Te(1)–S(1) 2.683(2) Å, Te(1)–S(1a) 2.553(2) Å for **3**, in this compound one of the bonds is significantly longer than the other, as was also found for  $Me_2Te[S_2P(OCMe_2)_2]_2$ ,  $Me_2Te[S_2P(OCH_2)_2CET_2]_2$  [15] and  $Ph_2Te[S_2P(OCH_2)_2CMe_2]_2$  [18]. The S–Te–S bond angles are of  $174.61(10)^\circ$  and  $174.08(8)^\circ$ , so that deviation from  $180^\circ$  is slightly less than in the dimethyltellurium xanthate and dithiocarbamate derivatives  $Me_2Te[S-COMe]_2$  [28] and  $Me_2Te[S_2CNMe_2]_2$  [7] where the an-

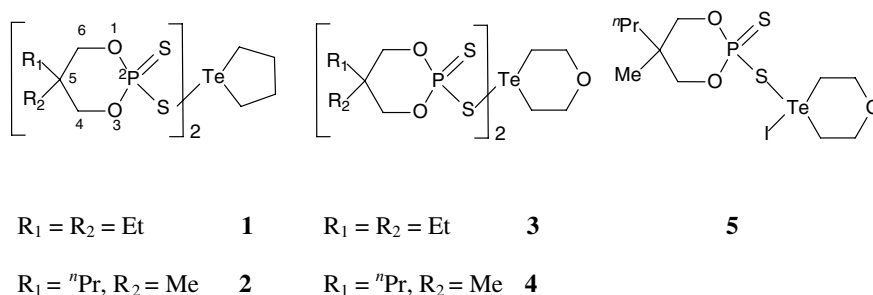
gles are  $165.90(3)^\circ$  and  $166.5(1)^\circ$ , respectively, and comparable to those reported for analogous non-cyclic dithiophosphates derivatives  $Ph_2Te[S_2P(OMe)_2]_2$  [10] and  $Ph_2Te[S_2P(OEt)_2]_2$  [8]. Table 3 presents some comparative values.

There are two longer secondary intramolecular Te–S interactions: Te(1)–S(2) 3.495 Å for **2** and Te(1)–S(2) 3.380 and 3.381 Å for **3**, respectively, these are substantially shorter than the sum of the van der Waals radii of 3.86 Å [27]. In comparison, the average Te–S secondary interactions in  $Ph_2Te[S_2P(OMe)_2]_2$  [10] and  $Ph_2Te[S_2P(OEt)_2]_2$  [8] are shorter than those in **2** and **3**, suggesting that the cyclic dithiophosphates are weaker chelating agents than non-cyclic dithiophosphates and both are weaker than dithiocarbamates and xanthates.

On the other hand, if the dithiophosphate groups are considered to be anisobidentate with the second sulfur atoms forming part of the coordination sphere, then the environment is bi-capped  $\psi$ -trigonal bipyramid, for **2**, in which the caps are in two faces opposite of the bipyramid and impart an overall seven coordinate geometry about the tellurium atom as in  $Ph_2Te[S_2P(OEt)_2]_2$  [8], while for **3**, the environment is a distorted octahedron with each of the anisobonding sulfur atoms taking up positions approximately *trans* to one of the methylenes attached to tellurium. Thus in compound **3**, S(1), S(2), Te(1) and S(1a) are approximately coplanar with the carbon atom attached to tellurium C(1a), out of the plane, similarly, S(2a), S(1a) Te(1) and S(1) are approximately coplanar with the carbon C(1) out of the plane. If these interactions Te–S may be regarded as secondary interactions, then these sulfur atoms would apparently complete a distorted octahedral configuration about the tellurium atom, which has been found in other organotellurium compounds [10].

The dithiophosphate bite angles of  $64.79(5)^\circ$  in **2** and  $65.79^\circ$  and  $68.05^\circ$  in **3** are comparable to those reported as for non-cyclic as cyclic dithiophosphate derivatives such as  $Ph_2Te[S_2P(OEt)_2]_2$  [8],  $Ph_2Te[S_2P(OMe)_2]_2$  [10] and  $Me_2Te[S_2P(OCMe_2)_2]_2$ ,  $Me_2Te[S_2P(OCH_2)_2CET_2]_2$  [15].

The Te–S–P bonds, which average 2.045(3) Å in **2** and 2.051(3) Å in **3**, are longer than the terminal P=S



Scheme 1.

Table 1

Crystallographic data for compound **2**, C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr]<sub>2</sub> and **3**, C<sub>4</sub>H<sub>8</sub>OTe[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt]<sub>2</sub>

	<b>2</b>	<b>3</b>
Compound formula	C <sub>18</sub> H <sub>36</sub> O <sub>4</sub> P <sub>2</sub> S <sub>4</sub> Te	C <sub>18</sub> H <sub>36</sub> O <sub>5</sub> P <sub>2</sub> S <sub>4</sub> Te
Formula weight	634.25	650.25
Crystal size (mm)	0.60 × 0.40 × 0.20	0.13 × 0.18 × 0.24
Wave length	0.710 73	0.710 73
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	P2 <sub>1</sub> /c
<i>a</i> (Å)	31.669(2)	15.710(2)
<i>b</i> (Å)	6.810(1)	6.9719(10)
<i>c</i> (Å)	12.733(2)	24.869(3)
α (°)	90	90
β (°)	90.13(1)	90.427(2)
γ (°)	90	90
<i>V</i> (Å <sup>3</sup> )	2746.1(6)	2723.9(7)
<i>Z</i>	4	4
<i>D</i> <sub>calc</sub> (mg m <sup>-3</sup> )	1.534	1.586
Absorption coefficient (mm <sup>-1</sup> )	1.525	1.542
<i>F</i> (000)	1288	1320
Temperature (K)	293(2)	293(2)
θ Range for data collection (°)	3.20 to 25.00	1.30 to 23.00
Range of <i>hkl</i>	0 ≤ <i>h</i> ≤ 37 0 ≤ <i>k</i> ≤ 8 −15 ≤ <i>l</i> ≤ 15	−17 ≤ <i>h</i> ≤ 17 −7 ≤ <i>k</i> ≤ 7 −27 ≤ <i>l</i> ≤ 27
Reflections collected	2308	20531
Independent reflections ( <i>R</i> <sub>int</sub> )	2263 (0.0438)	3799 (0.0638)
Maximum and minimum transmission	0.811 and 0.560	
Data/restraints/parameters	2263/0/142	3799/0/275
Goodness-of-fit	1.217	1.107
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0380, <i>wR</i> <sub>2</sub> = 0.1180	<i>R</i> <sub>1</sub> = 0.0589, <i>wR</i> <sub>2</sub> = 0.1081
Final indices (all data)	<i>R</i> <sub>1</sub> = 0.0490, <i>wR</i> <sub>2</sub> = 0.1651	<i>R</i> <sub>1</sub> = 0.0848, <i>wR</i> <sub>2</sub> = 0.1169
Largest difference peak and hole (e Å <sup>-3</sup> )	0.795 and −1.120	0.637 and −0.931

Table 2

Selected bond lengths (Å) and angles (°) for the compound **2**, C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe-<sup>n</sup>Pr]<sub>2</sub> and **3**, C<sub>4</sub>H<sub>8</sub>OTe[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt]<sub>2</sub>

	<b>2</b>	<b>3</b>
Te(1)–C(1)	2.133(7)	2.154(7)
Te(1)–C(1a)	2.133(7)	2.155(7)
Te(1)–S(1)	2.6410(18)	2.683(2)
Te(1)–S(1a)	2.6410(18)	2.553(2)
Te(1)–S(2)	3.495(2)	3.381
Te(1)–S(2a)	3.495(2)	3.380
S(1)–P(1)	2.045(3)	2.034(3)
S(2)–P(1)	1.929(2)	1.896(3)
S(1a)–P(1a)	2.045(3)	2.069(3)
S(2a)–P(1a)	1.929(2)	1.913(3)
C(1)–Te(1)–C(1a)	84.8(4)	93.0(3)
C(1a)–Te(1)–S(1)	84.8(4)	84.0(2)
C(1a)–Te(1)–S(1a)	90.7(2)	91.9(2)
C(1)–Te(1)–S(1)	90.7(2)	91.0(2)
S(1a)–Te(1)–S(1)	174.61(10)	174.08(8)
S(1)–Te(1)–S(2)	64.79(5)	67.79
S(1a)–Te(1)–S(2a)	64.79(5)	68.05
S(1)–P(1)–S(2)	115.75(11)	116.67(14)
S(1a)–P(1a)–S(2a)	115.75(11)	116.67(14)
P(1a)–S(1a)–Te(1)	96.43(8)	97.81(11)
O(2)–P(1)–O(1)	104.0(3)	103.5(3)
O(2a)–P(1a)–O(1a)	104.0(3)	104.4(3)

bonds, which average is 1.929(2) Å in **2** and 1.904(3) Å in **3**, the S–P–S bond angles average 115.75(11)° in **2** and 116.67(14)° in **3**. These distances and bond angles are comparable to those reported as for non-cyclic as cyclic dithiophosphates derivatives [11,13,18]. The O–P–O bond angles averaging 104.0(3)° in **2** and 103.95(3)° in **3** are similar to those reported for Me<sub>2</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt]<sub>2</sub> [14]. The bond lengths and angles within the ring systems are as expected.

In both structures **2** and **3**, the six-membered rings take up the chair conformation expected for a hexane ring. In **2**, the *n*-propyl group is in axial position on the ring and *trans* with respect to short bond S–Te and this is contrary to that found for this dithiophosphate ligand with other metals.

#### 4. Experimental

All reagents were of commercial grade and were used as received. The 1,1-diiodotellurane, C<sub>4</sub>H<sub>8</sub>TeI<sub>2</sub>, and 1-oxa-4,4-diiodo tellurane, C<sub>4</sub>H<sub>8</sub>OTeI<sub>2</sub>, were prepared according to the methods described by Ziolo and



Table 3  
Bond distances (Å) and angles (°) in some tellurium IV compounds

Compounds	Te–C	Te–S <sub>s</sub> <sup>a</sup>	Te–S <sub>l</sub> <sup>a</sup>	S <sub>s</sub> –Te–S <sub>s</sub>	S <sub>s</sub> –Te–S <sub>l</sub>	P–S	P=S	S <sub>s</sub> –P–S <sub>l</sub>	O–P–O	Reference
Ph <sub>2</sub> Te[S <sub>2</sub> P(OEt) <sub>2</sub> ] <sub>2</sub>	2.130	2.609	3.367	170.56	66.58	2.043	1.931	114.42	94.8	[8]
		2.620	3.353		66.88	2.020	1.929	115.92	96.2	
Ph <sub>2</sub> Te[S <sub>2</sub> P(OMe) <sub>2</sub> ] <sub>2</sub>	2.133	2.619	3.383	171.8	66.7	2.045	1.934	115.1	94.8	[10]
	2.136	2.625	3.362		66.9	2.022	1.928	116.3	93.6	
Me <sub>2</sub> Te[S <sub>2</sub> P(OCMe) <sub>2</sub> ] <sub>2</sub>	2.127	2.127	3.349	173.26	66.86	2.045	1.939	113.8	97.1	[15]
	2.117	2.117	3.341		66.31	2.038	1.944	113.6	97.0	
Me <sub>2</sub> Te[S <sub>2</sub> P(OCH <sub>2</sub> ) <sub>2</sub> CEt <sub>2</sub> ] <sub>2</sub>	2.113	2.597	3.490	169.52	65.56	2.060	1.917	116.4	104.1	[15]
	2.128	2.660	3.516		64.66	2.052	1.920	116.7	103.5	
Ph <sub>2</sub> Te[S <sub>2</sub> P(OCMe <sub>2</sub> ) <sub>2</sub> ] <sub>2</sub>	2.127	2.636	3.323	173.78	67.22	2.047	1.938	114.3	96.8	[18]
	2.132	2.624	3.402		66.27	2.055	1.921	115.2	97.1	
Ph <sub>2</sub> Te[S <sub>2</sub> P(OCH <sub>2</sub> ) <sub>2</sub> CMe <sub>2</sub> ] <sub>2</sub>	2.15	2.657	3.637	168.3	62.4	2.033	1.920	116.8	104.3	[18]
	2.12	2.615	3.479		65.1	2.052	1.901	116.3	103.4	
C <sub>4</sub> H <sub>8</sub> Te[S <sub>2</sub> P(OCH <sub>2</sub> ) <sub>2</sub> CMe <sup>n</sup> Pr] <sub>2</sub>	2.133	2.641	3.495	174.61	64.79	2.045	1.929	115.75	104.0	This work
	2.133	2.641	3.495		64.79	2.045	1.929	115.75	104.0	
C <sub>4</sub> H <sub>8</sub> OTe[S <sub>2</sub> P(OCH <sub>2</sub> ) <sub>2</sub> CEt <sub>2</sub> ] <sub>2</sub>	2.154	2.683	3.381	174.08	67.79	2.034	1.896	116.67	103.5	This work
	2.155	2.553	3.380		68.05	2.069	1.913	116.67	104.4	

<sup>a</sup> S<sub>s</sub>, sulfur short distance; S<sub>l</sub> sulfur large distance.

Gunther [29]. The sodium salts of cyclic dithiophosphate were prepared according to the reported literature methods [21].

IR spectra were recorded in the region 4000–500 cm<sup>-1</sup> as KBr pellets using Bruker spectrometer. The <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P and <sup>125</sup>Te magnetic resonance spectra were obtained on a Varian Gemini spectrometer of 200 and 400 MHz at room temperature in CDCl<sub>3</sub>. The chemical shifts are relative to internal Me<sub>4</sub>Si (<sup>1</sup>H, <sup>13</sup>C) and external H<sub>3</sub>PO<sub>4</sub> 85% (<sup>31</sup>P) and 1,1-diiodotelluroane (<sup>125</sup>Te) for the indicated nuclei. Mass spectra determinations were performed on a JEOL JMS-WSX 102A instrument by electron impact at 70 eV.

Single crystals of **2** and **3** suitable for X-ray diffraction were obtained by diffusion using a dichloromethane–*n*-hexane mixture. Crystallographic and structure solution data are summarized in Table 1. Data collections were carried out in a Siemens P4/PC diffractometer at room temperature, with graphite-monochromated Mo Kα radiation (λ = 0.71073). The structures were solved by direct methods using SHELXTL program. PLUS(PC version) for **2**. For structure **3**, the data collections were carried out in a Bruker APEX CCD diffractometer at room temperature.

#### 4.1. Synthesis of telluroane-1,1-diyl bis (5',5'-diethyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate)

##### C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt<sub>2</sub>]<sub>2</sub> (**1**)

This was prepared by the reaction of C<sub>4</sub>H<sub>8</sub>TeI<sub>2</sub> (0.3550 g, 0.8115 mmol) with Na[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CEt<sub>2</sub>] (0.4026 g, 1.623 mmol) in chloroform (30 ml). The solution was stirred for 2 h, filtered off and evaporated to dryness. The resulting solid was recrystallized from a

dichloromethane–*n*-hexane mixture. White solid, m.p. 148–150 °C. Yield (0.216 g, 42%). EI MS (70 eV) *m/z* 376 (C<sub>4</sub>H<sub>8</sub>S<sub>4</sub>P<sub>2</sub>Te<sup>+</sup>, 18%), 352 (C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>S<sub>2</sub>PTe<sup>+</sup>, 75%), 281 (C<sub>4</sub>H<sub>8</sub>S<sub>2</sub>PTe<sup>+</sup>, 15%), 225 (C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>S<sub>2</sub>P<sup>+</sup>, <10%), 186 (C<sub>4</sub>H<sub>8</sub>Te<sup>+</sup>, <10%). IR (KBr) 2970 m, 2939 m, 2920 m, 2878 m ν(C–H), 1186 w ν(C–O), 1072 s, 1019 s ν((P)–O–C), 880 s ν(P–O–(C)), 994 s, 935 m ν(P–O<sub>2</sub>), 670 s ν(P=S), 516 s ν(P–S) cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 0.85 (12H, t, CH<sub>2</sub>CH<sub>3</sub>), 1.47 (8H, q, CH<sub>2</sub>CH<sub>3</sub>), 2.81 (4H, bs, CH<sub>2</sub>CH<sub>2</sub>Te), 3.55 (4H, bs, CH<sub>2</sub>Te), 4.11 (8H, d, O–CH<sub>2</sub>–C, <sup>3</sup>J<sub>P–H</sub> = 16 Hz). <sup>13</sup>C NMR (CDCl<sub>3</sub>): δ 7.29 (s, CH<sub>2</sub>CH<sub>3</sub>), 23.34 (s, CH<sub>2</sub>CH<sub>3</sub>), 33.91 (s, CH<sub>2</sub>CH<sub>2</sub>Te), 37.65 (d, OCH<sub>2</sub>CCH<sub>2</sub>, <sup>3</sup>J<sub>CP</sub> = 5.4 Hz), 45.36 (s, CH<sub>2</sub>Te), 73.96 (d, P–O–CH<sub>2</sub>–C, <sup>2</sup>J<sub>CP</sub> = 8.4 Hz). <sup>31</sup>P NMR (CDCl<sub>3</sub>): δ 94.20 (s). <sup>125</sup>Te NMR (CDCl<sub>3</sub>): δ 851.87.

#### 4.2. Synthesis of tellurole-1,1-diyl bis(5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate)

##### C<sub>4</sub>H<sub>8</sub>Te[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe<sup>n</sup>Pr]<sub>2</sub> (**2**)

This compound was prepared in a similar manner to **1** from C<sub>4</sub>H<sub>8</sub>TeI<sub>2</sub> (0.2644 g, 0.6044 mmol) with Na[S<sub>2</sub>P(OCH<sub>2</sub>)<sub>2</sub>CMe<sup>n</sup>Pr] (0.2999 g, 1.2088 mmol). The resulting white solid was recrystallized from a dichloromethane–*n*-hexane mixture. This solid, m.p. 145–147 °C. Yield (0.109 g, 29%). Suitable crystals for X-ray diffraction were grown by allowing a layer of *n*-hexane to diffuse slowly into a solution of **2** in dichloromethane at room temperature. EI MS (70 eV) *m/z* 368 (C<sub>8</sub>H<sub>15</sub>O<sub>2</sub>S<sub>2</sub>PTe<sup>+</sup>, <10%), 322 (C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>SPTe<sup>+</sup>, >15%), 191 (P<sub>2</sub>S<sub>4</sub><sup>+</sup>, >25%), 181 (C<sub>4</sub>H<sub>6</sub>O<sub>2</sub>S<sub>2</sub>P<sup>+</sup>, 75%), 150 (C<sub>4</sub>H<sub>6</sub>O<sub>2</sub>SP<sup>+</sup>, 25%). IR (KBr) 2959 m, 2938 m, 2872 m ν(C–H), 1195 w ν(C–O), 1057 m, 1041 m, 1017 m

$\nu(\text{P}-\text{O}-\text{C})$ , 814 s, 805 s  $\nu(\text{P}-\text{O}-(\text{C}))$ , 986 s, 959 s  $\nu(\text{P}-\text{O}_2)$ , 684 s, 672 s  $\nu(\text{P}=\text{S})$ , 515 m  $\nu(\text{P}-\text{S})$   $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  0.94 (6H, t,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.01 (6H, s,  $\text{CH}_3$ ), 1.30 (4H, com  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.40 (4H, com  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 2.81 (4H, bs,  $\text{CH}_2\text{CH}_2\text{Te}$ ), 3.55 (4H, bs,  $\text{CH}_2\text{Te}$ ), 4.07 (8H, ddd,  $\text{O}-\text{CH}_2-\text{C}$ ,  $J_{\text{gem}} = 11$  Hz,  $^3J_{\text{P}-\text{H}} = 15.6$  Hz).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  14.88 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 16.59 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 19.07 (s,  $\text{CH}_3$ ), 33.92 (s,  $\text{CH}_2\text{CH}_2\text{Te}$ ), 35.39 (d,  $\text{OCH}_2\text{CCH}_2$ ,  $^3J_{\text{CP}} = 6.13$  Hz), 37.08 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 45.35 (s,  $\text{CH}_2\text{Te}$ ), 75.58 (d,  $\text{P}-\text{O}-\text{CH}_2-\text{C}$ ,  $^2J_{\text{CP}} = 8.4$  Hz).  $^{31}\text{P}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  93.62 (s).  $^{125}\text{Te}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  851.72.

**4.3. Synthesis of 1-oxa-4-tellurane-4,4-diyl bis (5',5'-diethyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate)  $\text{C}_4\text{H}_8\text{OTe}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CET}_2]_2$  (**3**)**

This was prepared by the reaction of  $\text{C}_4\text{H}_8\text{OTeI}_2$  (0.30 g, 0.661 mmol) with  $\text{Na}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CET}_2]$  (0.3283 g, 1.323 mmol) in ethanol (30 ml). The solution was stirred for 24 h. The precipitated pale yellow solid was filtered, washed with ethanol and dried in vacuo. The resulting solid was recrystallized from a dichloromethane-*n*-hexane mixture. This solid m.p. 155 °C. Yield (0.238 g, 56%). Suitable crystals for X-ray diffraction were grown by allowing a layer of *n*-hexane to diffuse slowly into a solution of **3** in dichloromethane at room temperature. EI MS (70 eV)  $m/z$  450 ( $\text{C}_{14}\text{H}_{28}\text{O}_4\text{S}_4\text{P}_2^+$ , 63%), 202 ( $\text{C}_4\text{H}_8\text{OTe}^+$ , 10%), 193 ( $\text{C}_7\text{H}_{14}\text{O}_2\text{SP}^+$ , 15%), 161 ( $\text{C}_7\text{H}_{14}\text{O}_2\text{P}^+$ , 100%). IR (KBr) 2967 m, 2935 m, 2873 m  $\nu(\text{C}-\text{H})$ , 1186 w, 1161 w, 1271 w  $\nu(\text{C}-\text{O})$ , 1067 m, 1017 s  $\nu(\text{P}-\text{O}-\text{C})$ , 808 s  $\nu(\text{P}-\text{O}-(\text{C}))$ , 991 s, 935 m  $\nu(\text{P}-\text{O}_2)$ , 673 s  $\nu(\text{P}=\text{S})$ , 507 m  $\nu(\text{P}-\text{S})$   $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  0.86 (12H, t,  $\text{CH}_2\text{CH}_3$ ), 1.48 (8H, q,  $\text{CH}_2\text{CH}_3$ ), 3.35 (4H, t,  $\text{CH}_2\text{Te}$ ), 4.13 (8H, d,  $\text{O}-\text{CH}_2-\text{C}$ ,  $^3J_{\text{P}-\text{H}} = 16$  Hz), 4.27 (4H, t,  $\text{O}-\text{CH}_2\text{CH}_2\text{Te}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  7.27 (s,  $\text{CH}_2\text{CH}_3$ ), 23.22 (s,  $\text{CH}_2\text{CH}_3$ ), 29.69 (s,  $\text{CH}_2\text{CH}_2\text{Te}$ ), 37.61 (d,  $\text{CH}_2\text{CCH}_2$ ,  $^3J_{\text{CP}} = 5.9$  Hz), 65.43 (s,  $\text{O}-\text{CH}_2\text{CH}_2$ ), 74.13 (d,  $\text{P}-\text{O}-\text{CH}_2-\text{C}$ ,  $^2J_{\text{CP}} = 7.3$  Hz).  $^{31}\text{P}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  92.54 (s) ( $J_{\text{PH}} = 14.81$  Hz).  $^{125}\text{Te}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  557.33.

**4.4. Synthesis of 1-oxa-4-tellurane-4,4-diyl bis (5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate)  $\text{C}_4\text{H}_8\text{OTe}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}]_2$  (**4**)**

This was prepared by the reaction of  $\text{C}_4\text{H}_8\text{OTeI}_2$  [ $\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}$ ] (**5**) (0.100 g, 0.1812 mmol) with  $\text{Na}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}]$  (0.0449 g, 0.1812 mmol) in chloroform (30 ml). The solution was stirred for 24 h, filtered off and evaporated. The resulting white solid was recrystallized from a dichloromethane-*n*-hexane mixture. This solid m.p. 107–110 °C. Yield (0.070 g, 60%). EI MS (70 eV)  $m/z$  652 ( $\text{M}^+$ , <10%), 427

( $\text{C}_{11}\text{H}_{22}\text{O}_3\text{S}_2\text{P}^+$ , 100%), 411 ( $\text{C}_{11}\text{H}_{22}\text{O}_2\text{S}_2\text{P}^+$ , <10%), 355 ( $\text{C}_7\text{H}_{14}\text{O}_2\text{S}_2\text{P}^+$ , <10%), 225 ( $\text{C}_7\text{H}_{14}\text{O}_2\text{S}_2\text{P}^+$ , 10%), 202 ( $\text{C}_4\text{H}_8\text{OTe}^+$ , <10%). IR (KBr) 2960 m, 2932 m, 2875 m  $\nu(\text{C}-\text{H})$ , 1272 w, 1197 w  $\nu(\text{C}-\text{O})$ , 1089 w, 1055 m, 1014 m  $\nu(\text{P}-\text{O}-\text{C})$ , 813 s  $\nu(\text{P}-\text{O}-(\text{C}))$ , 987 s  $\nu(\text{P}-\text{O}_2)$ , 677 s, 612 w  $\nu(\text{P}=\text{S})$ , 516 m  $\nu(\text{P}-\text{S})$   $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  0.94 (6H, t,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.02 (6H, s,  $-\text{CH}_3$ ), 1.32 (4H, com,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.40 (4H, com,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 3.35 (4H, s,  $\text{CH}_2\text{Te}$ ), 4.10 (8H, dd,  $\text{O}-\text{CH}_2-\text{C}$ ,  $^3J_{\text{P}-\text{H}} = 15.60$  Hz  $^1J_{\text{gem}} = 11.20$  Hz), 4.27 (4H, s,  $\text{O}-\text{CH}_2\text{CH}_2\text{Te}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  14.88 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 16.56 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 19.01 (s,  $\text{CH}_3$ ), 29.66 (sb,  $\text{CH}_2\text{CH}_2\text{Te}$ ), 35.31 (s,  $\text{OCH}_2\text{CCH}_2$ ), 36.96 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 65.42 (s,  $\text{OCH}_2\text{CH}_2\text{Te}$ ), 75.71 (d,  $\text{P}-\text{O}-\text{CH}_2-\text{C}$ ,  $^2J_{\text{CP}} = 7.99$  Hz).  $^{31}\text{P}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  91.97 (s).  $^{125}\text{Te}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  565.89.

**4.5. Synthesis of 1-oxa-4-tellurane-4-iodide-4-yl-(5'-methyl-5'-*n*-propyl-2'-thiono-1',3',2'-dioxaphosphinane-2'-thiolate)  $\text{C}_4\text{H}_8\text{OTeIS}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}$ ] (**5**)**

This compound was prepared in a similar manner to **3**, from  $\text{C}_4\text{H}_8\text{OTeI}_2$  (0.300 g, 0.662 mmol) with  $\text{Na}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}]$  (0.3283 g, 1.323 mmol) in ethanol (30 ml). The solution was stirred for 24 h. Even though the reactions were prepared in the molar ratio 1:2, the compound obtained was monosubstituted  $\text{C}_4\text{H}_8\text{OTe}[\text{S}_2\text{P}(\text{OCH}_2)_2\text{CMe}^n\text{Pr}]$  (**5**).

The resulting pale yellow solid was filtered and recrystallized from an acetone-*n*-hexane mixture. This solid m.p. 126–128 °C. Yield (0.254 g, 59%). EI MS (70 eV)  $m/z$  450 ( $\text{C}_{14}\text{H}_{28}\text{O}_4\text{S}_4\text{P}_2^+$ , 10%), 427 ( $\text{C}_{14}\text{H}_{28}\text{O}_4\text{S}_4\text{P}_2^+$ , <10%), 384 ( $\text{C}_8\text{H}_{15}\text{O}_3\text{S}_2\text{P}^+$ , <10%), 329 ( $\text{C}_4\text{H}_8\text{OTeI}^+$ , <10%), 254 ( $\text{I}_2$ , 100%), 202 ( $\text{C}_4\text{H}_8\text{OTe}^+$ , 42%). IR (KBr) 2959 m, 2929 m, 2868 m  $\nu(\text{C}-\text{H})$ , 1269 w, 1196 w, 1164 w  $\nu(\text{C}-\text{O})$ , 1087 m, 1052 w, 1012 m  $\nu(\text{P}-\text{O}-\text{C})$ , 815 m  $\nu(\text{P}-\text{O}-(\text{C}))$ , 983 s, 949 m, 888 w  $\nu(\text{P}-\text{O}_2)$ , 674 s, 611 w  $\nu(\text{P}=\text{S})$ , 509 m  $\nu(\text{P}-\text{S})$   $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  0.94 (3H, t,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.02 (3H, s,  $-\text{CH}_3$ ), 1.32 (2H, com,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 1.40 (2H, com,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 3.35 (4H, b,  $\text{CH}_2\text{Te}$ ), 4.10 (4H, dd,  $\text{O}-\text{CH}_2-\text{C}$ ,  $^3J_{\text{P}-\text{H}} = 15.60$  Hz  $^1J_{\text{gem}} = 11.20$  Hz), 4.41 (4H, b,  $\text{O}-\text{CH}_2\text{CH}_2\text{Te}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  14.87 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 16.56 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 18.96 (s,  $\text{CH}_3$ ), 27.56 (sb,  $\text{CH}_2\text{CH}_2\text{Te}$ ), 35.38 (s,  $\text{OCH}_2\text{CCH}_2$ ), 36.92 (s,  $\text{CH}_2\text{CH}_2\text{CH}_3$ ), 65.12 (sb,  $\text{OCH}_2\text{CH}_2\text{Te}$ ), 75.74 (d,  $\text{P}-\text{O}-\text{CH}_2-\text{C}$ ,  $^2J_{\text{CP}} = 8.84$  Hz).  $^{31}\text{P}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  92.43 (s).  $^{125}\text{Te}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  564.75

## 5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic

Data Centre, CCDC 260870 for compound **2** and CCDC 260871 for compound **3**.

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