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Synthesis, spectroscopic characterisation of 1,1,2,3,4,5-hexahydro-1,1-dicarboxylatotellurophenes and crystal structures of 1,1,2,3,4,5-hexahydro-1,1-di(benzoato)- and 1,1-di(4-nitrobenzoato)tellurophene

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Abstract

1,1,2,3,4,5-hexahydro-1,1-dicarboxylatotellurophenes $C_4H_8Te(OCOR)_2$ ($R = CH_2Cl$, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$) were obtained from the reactions of 1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene with silver oxide and carboxylic acids or silver carboxylates. They were characterised by IR and (^1H, ^{13}C, ^{125}Te)-NMR spectroscopy. The structures of $C_4H_8Te(OCOC_6H_5)_2$ and $C_4H_8Te(4\text{-}NO_2C_6H_4OCO)_2$ were established by single-crystal X-ray diffraction studies. In both cases the coordination geometry about the central Te atom can be described as pseudotrigonal bipyramidal in which two oxygen atoms of the unidentate carboxylate groups are in the axial positions, two methylene carbon atoms (attached to Te) of the C_4H_8 group and the stereochemically active electron lone pair occupy the equatorial positions. The molecules are packed in their unit cells as the weakly bridged dimers through intermolecular Te···O secondary bonds.

Keywords: Tellurium; Carboxylate; Secondary bonds

1. Introduction

1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene (C_4H_8-TeI_2) was synthesised as early as 1931 [1]. Its derivatives and anionic complexes [2,3] have been reported from our laboratory including 1,1,2,3,4,5-hexahydro-1,-1-dicarboxylatotellurophenes viz. $C_4H_8Te(OCOR)_2$ ($R = CH_3$, CCl_3, C_6H_5) [2]. These dicarboxylate derivatives were assigned probable geometry on the basis of spectroscopic (IR, ^1H-NMR) characteristics, but there has not yet been crystallographic confirmation of this. To our knowledge, in fact, there is only one cyclic tellurium dicarboxylate viz. 10,10-di(trifluoroacetato)telluroxane [4] known, whose crystal structure has been determined.

No structural study on 1,1,2,3,4,5-hexahydrotellurophene derivatives in general (1,1,2,3,4,5-hexahydro-1,1-dicarboxylatotellurophene in particular) has so far been reported. In continuation of our structural studies on hypervalent Te(IV) compounds [5] and acyclic diorgano tellurium dicarboxylates [6] in the present investigation, we report on the synthesis and characterisation of $C_4H_8Te(OCOR)_2$ ($R = CH_2Cl, C_6H_5$, 4-NO_2-C_6H_4-3,5-$(NO_2)_2C_6H_3$-4-$OCH_3C_6H_4$) and X-ray structures of 1,1,2,3,4,5-hexahydro-1,1-di(benzoato)- and 1,1-di(4-nitrobenzoato)tellurophenes.

2. Experimental

2.1. Physical measurements

Elemental analyses for C, H and N were carried out on an Elemental Analyser Heraeus Carlo Erba 1108. IR spectra were recorded using a Shimadzu 8210 PC FTIR spectrometer in the frequency range 4000–350 cm^{-1} with the samples in KBr discs. The (^1H, ^{13}C, ^{125}Te)-

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NMR spectra were recorded on a Varian VXR-3005 spectrometer in $CDCl_3$ for all the compounds except 1,1,2,3,4,5-hexahydro-1,1-di(3,5-dinitrobenzoato) tellurophene (**4**), which was recorded in acetone. The operating frequency for ^{125}Te-NMR was 94.752 MHz with a pulse width of 9.5 μs and a delay of 1 s. ^{125}Te-NMR spectra were referenced to Me_2Te ($\delta = 0$ ppm). The single-crystal X-ray diffraction studies were carried out at the Chemistry Department, Howard University, Washington, DC.

Table 1
Crystal data and refinement details for compounds **2** and **3**

	2	**3**
Empirical formula	$C_{18}H_{18}O_4Te$	$C_{18}H_{16}N_2O_8Te$
Formula weight	425.92	515.93
Temperature (K)	293(2)	293(2)
Crystal system	Monoclinic	Triclinic
Space group	$C2/c$	$P1$
a (Å)	18.765(2)	6.2813(11)
b (Å)	10.7770(11)	11.534(2)
c (Å)	8.7490(8)	14.392(2)
α (°)	90	69.289(9)
β (°)	105.003(7)	88.238(14)
γ (°)	90	89.792(13)
V (Å^3)	1709.0(3)	974.9(3)
Z	4	2
μ (mm^{-1})	1.757	1.575
Index ranges	$0 \le h \le 22$, $0 \le k \le 14$, $-11 \le l \le 11$	$0 \le h \le 8$, $-14 \le k \le 14$, $-18 \le l \le 18$
Reflections collected	2103	4821
Independent reflections	2043 [$R_{int} = 0.0436$]	4414 [$R_{int} = 0.0172$]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0563$, $wR_2 = 0.1314$	$R_1 = 0.0332$, $wR_2 = 0.0777$
R indices (all data)	$R_1 = 0.0585$, $wR_2 = 0.1343$	$R_1 = 0.0427$, $wR_2 = 0.0831$

Table 2
Bond lengths (Å) and bond angles (°) for **2** [a]

Te–C(11) # 1	2.119(4)	Te–C(11)	2.119(4)
Te–O(1)	2.166(3)	Te–O(1) # 1	2.166(3)
O(1)–C(7)	1.303(5)	O(2)–C(7)	1.213(5)
C(11)–C(12)	1.524(6)	C(12)–C(12) # 1	1.514(9)
C(1)–C(6)	1.393(6)	C(1)–C(2)	1.398(5)
C(1)–C(7)	1.493(6)	C(2)–C(3)	1.382(6)
C(3)–C(4)	1.385(8)	C(4)–C(5)	1.392(7)
C(5)–C(6)	1.368(6)		
C(11) # 1–Te–C(11)	85.5(2)	C(11) # 1–Te–O(1)	83.6(2)
C(11)–Te–O(1)	81.83(14)	C(11) # 1–Te–O(1) # 1	81.83(14)
C(11)–Te–O(1) # 1	83.6(2)	O(1)–Te–O(1) # 1	160.1(2)
C(7)–O(1)–Te	114.1(3)	C(12)–C(11)–Te	105.5(3)
C(12) # 1–C(12)–C(11)	109.8(3)	C(6)–C(1)–C(2)	118.9(4)
C(6)–C(1)–C(7)	122.2(4)	C(2)–C(1)–C(7)	118.9(3)
C(3)–C(2)–C(1)	119.9(4)	C(2)–C(3)–C(4)	120.7(4)
C(3)–C(4)–C(5)	119.3(4)	C(6)–C(5)–C(4)	120.3(4)
C(5)–C(6)–C(1)	120.9(4)	O(2)–C(7)–O(1)	122.8(4)
O(2)–C(7)–C(1)	122.6(4)	O(1)–C(7)–C(1)	114.6(3)

[a] Symmetry transformations used to generate equivalent atoms: # 1 $-x+1$, y, $-z+1/2$.

Table 3
Bond lengths (Å) and bond angles (°) for **3**

Te–C(11)	2.085(4)	Te–C(14)	2.109(4)
Te–O(1B)	2.167(2)	Te–O(1A)	2.178(2)
O(1A)–C(1A)	1.297(4)	O(2A)–C(1A)	1.220(4)
O(3A)–N(1A)	1.213(4)	O(4A)–N(1A)	1.219(4)
O(1B)–C(1B)	1.306(4)	O(2B)–C(1B)	1.223(4)
O(3B)–N(1B)	1.213(5)	O(4B)–N(1B)	1.215(5)
N(1A)–C(5A)	1.473(4)	N(1B)–C(5B)	1.482(4)
C(11)–C(12)	1.472(9)	C(12)–C(13)	1.466(10)
C(13)–C(14)	1.519(8)	C(1A)–C(2A)	1.494(4)
C(2A)–C(3A)	1.392(4)	C(2A)–C(7A)	1.400(4)
C(3A)–C(4A)	1.377(4)	C(4A)–C(5A)	1.383(4)
C(5A)–C(6A)	1.382(4)	C(6A)–C(7A)	1.369(4)
C(1B)–C(2B)	1.498(4)	C(2B)–C(7B)	1.388(4)
C(2B)–C(3B)	1.396(4)	C(3B)–C(4B)	1.374(5)
C(4B)–C(5B)	1.380(5)	C(5B)–C(6B)	1.376(5)
C(6B)–C(7B)	1.375(4)		
C(11)–Te–C(14)	86.6(2)	C(11)–Te–O(1B)	83.69(13)
C(14)–Te–O(1B)	88.10(12)	C(11)–Te–O(1A)	83.04(13)
C(14)–Te–O(1A)	88.90(12)	O(1B)–Te–O(1A)	166.54(8)
C(1A)–O(1A)–Te	111.7(2)	C(1B)–O(1B)–Te	109.0(2)
O(3A)–N(1A)–O(4A)	123.1(3)	O(3A)–N(1A)–C(5A)	118.7(3)
O(4A)–N(1A)–C(5A)	118.1(3)	O(3B)–N(1B)–O(4B)	123.8(3)
O(3B)–N(1B)–C(5B)	117.5(4)	O(4B)–N(1B)–C(5B)	118.7(3)
C(12)–C(11)–Te	105.7(3)	C(13)–C(12)–C(11)	114.6(5)
C(12)–C(13)–C(14)	112.4(5)	C(13)–C(14)–Te	104.6(3)
O(2A)–C(1A)–O(1A)	122.5(3)	O(2A)–C(1A)–C(2A)	122.0(3)
O(1A)–C(1A)–C(2A)	115.6(3)	C(3A)–C(2A)–C(7A)	119.3(3)
C(3A)–C(2A)–C(1A)	119.4(3)	C(7A)–C(2A)–C(1A)	121.3(3)
C(4A)–C(3A)–C(2A)	120.8(3)	C(3A)–C(4A)–C(5A)	118.3(3)
C(6A)–C(5A)–C(4A)	122.2(3)	C(6A)–C(5A)–N(1A)	119.1(3)
C(4A)–C(5A)–N(1A)	118.7(3)	C(7A)–C(6A)–C(5A)	119.0(3)
C(6A)–C(7A)–C(2A)	120.3(3)	O(2B)–C(1B)–O(1B)	123.2(3)
O(2B)–C(1B)–C(2B)	121.5(3)	O(1B)–C(1B)–C(2B)	115.3(3)
C(7B)–C(2B)–C(3B)	119.5(3)	C(7B)–C(2B)–C(1B)	122.2(3)
C(3B)–C(2B)–C(1B)	118.3(3)	C(4B)–C(3B)–C(2B)	120.2(3)
C(3B)–C(4B)–C(5B)	118.6(3)	C(6B)–C(5B)–C(4B)	122.8(3)
C(6B)–C(5B)–N(1B)	118.4(3)	C(4B)–C(5B)–N(1B)	118.8(3)
C(7B)–C(6B)–C(5B)	118.0(3)	C(6B)–C(7B)–C(2B)	120.9(3)

2.2. Synthesis

1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene (C_4H_8-TeI_2) was prepared by the literature method [1] and recrystallised from benzene. $C_4H_8TeI_2$ (2 g, 4.57 mmol), silver oxide (1.06 g, 4.57 mmol) and monochloroacetic acid (0.86 g, 9.14 mmol) were stirred in acetone (ca. 30 ml) for 4 h. The reaction mixture was filtered and the filtrate concentrated under reduced pressure to give 1,1,2,3,4,5 - hexahydro - 1,1 - di(monochloroacetato)tellurophene (**1**). Compound **1** yield: 1.2 g (71%), m.p. 100°C. Anal. Calc. for $C_8H_{12}O_4Cl_2Te$: C, 25.90; H, 3.24; Te, 34.43. Found: C, 25.82; H, 3.20; Te, 34.40%.

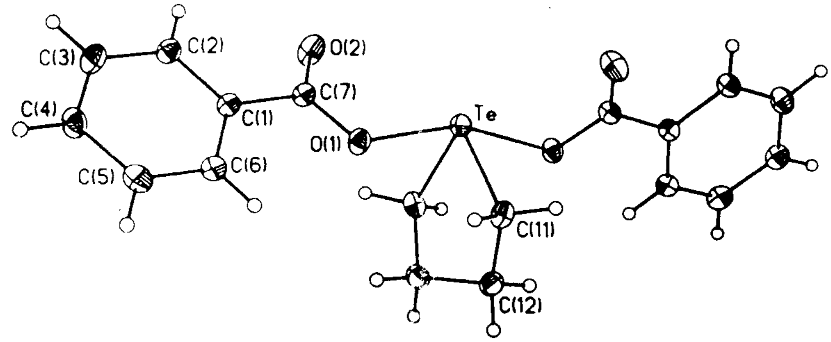

Fig. 1. Crystal structure of **2**.

1,1,2,3,4,5 - hexahydro - 1,1 - di(benzoato)tellurophene (**2**); 1,1,2,3,4,5-hexahydro-1,1-di(4-nitrobenzoato)tellurophene (**3**); 1,1,2,3,4,5-hexahydro-1,1-di(3,5-dinitrobenzoato)tellurophene (**4**) and 1,1,2,3,4,5-hexahydro-1,1-di(4-methoxybenzoato)tellurophene (**5**) were prepared by stirring $C_4H_8TeI_2$ with freshly prepared silver carboxylates in acetone.

$$C_4H_8TeI_2 + 2AgOCOR \rightarrow C_4H_8Te(OCOR)_2 \quad (1)$$

$$(R = C_6H_5\text{-}4NO_2C_6H_4\text{-}3,5\text{-}(NO_2)_2C_6H_3\text{-}4\text{-}OCH_3C_6H_4)$$

To 1,1,2,3,4,5-hexahydro-1,1-diiodotellurophene (2 g, 4.57 mmol) was added freshly prepared silver benzoate (2.10 g, 9.17 mmol), silver 4-nitrobenzoate (2.51 g, 9.16 mmol), silver 3,5-dinitrobenzoate (2.92 g, 9.15 mmol), silver 4-methoxybenzoate (2.37 g, 9.15 mmol) in acetone (ca. 30 ml). It was stirred for 4 h and filtered to eliminate AgI and excess silver carboxylates. The filtrate was concentrated under reduced pressure to give compounds **2**–**5**. Compound **2** yield: 1.45 g (75%), m.p. 145°C. Anal. Calc. for $C_{18}H_{18}O_4Te$: C, 50.75; H, 4.23; Te, 29.98. Found: C, 50.70; H, 4.15; Te, 29.92%. Compound **3** yield: 1.41 g (60%), m.p. 220°C. Anal. Calc. for $C_{18}H_{16}N_2O_8Te$: C, 41.89; H, 3. 10; N, 5.43; Te, 24.75. Found: C, 41.75; H, 3.05; N, 5.40; Te, 24.68%. Compound **4** yield: 1 g (36%), m.p. 230°C. Anal. Calc. for $C_{18}H_{14}N_4O_2Te$: C, 35.67; H, 2.31; N, 9.25; Te, 21.07. Found: C, 35.62; H, 2.25; N, 9. 12; Te, 21.00%. Compound **5** yield: 1.56 g (70%) m.p. 200°C. Anal. Calc. for $C_{20}H_{22}O_6Te$: C, 49.42; H, 4.53; Te, 26.28. Found: C, 49.36; H, 4.52; Te, 26.22%.

2.3. X-ray measurements

The X-ray measurements for compounds **2** and **3** were performed at 293(2) K. The structure of **2** was solved in space group $C2/c$ and that of **3** in space group $P1$. The colourless crystals ($0.16 \times 0.67 \times 0.12$ mm) of compound **2** and pale yellow crystals ($0.10 \times 0.99 \times 0.16$ mm) of compound **3** were mounted on a Siemens R3m/v diffractometer using graphite monochromated Mo–K_α radiation (0.71073 Å). The unit cells were determined from 25 randomly selected reflections using the automatic search index and least-square routine. The data collected in the θ range from 3.04 to 27.99° for **2** and from 2.81 to 28.57° for **3** correspond to monoclinic and triclinic cells, respectively whose dimensions are given in Table 1. The data were corrected for Lorentz, polarization and absorption effects. The maximum and minimum transmission values of the correction factor are 0.5123 and 0.3208. The data were monitored by measuring two standard reflections every 60 min of X-ray exposure time. The structure was solved by routine heavy atom using SHELXS-86 [7] and Fourier methods and refined by full-matrix least-squares with the non hydrogen atoms anisotropic at hydrogens having fixed isotropic thermal parameters of 0.07 $Å^2$ using the SHELXL-93 program [8]. Selected bond lengths and bond angles for **2** and **3** are listed in Tables 2 and 3, respectively. The crystal structures of **2** and **3** are shown in Fig. 1 and Fig. 4; the packing diagram of **2** is shown in Fig. 3.

3. Results and discussion

3.1. Spectroscopic characterisation

Spectroscopic data for **1**–**5** are given in Table 5. The IR spectra of **1**–**5** display the characteristic carboxylate group frequencies. The ν asym COO and ν sym COO are in the range 1617–1670 and 1321–1387 cm^{-1},

Table 4
Bond lengths and angles for **2** in $C2/c$

TE	O1	2.166					
TE	C11	2.119	81.8				
TE	O1A	2.166	160.1	83.6			
TE	C11A	2.119	83.6	85.5	81.8		
TE	O2B	3.083	113.1	149.4	74.5	70.6	
TE	O2C	3.083	74.5	70.6	113.1	149.4	137.8
			O1	C11	O1A	C11A	O2B

Table 5
Spectroscopic data for **1**–**5**

Compound	IR (KBr) (cm^{-1})	^{1}H-NMR ($CDCl_3$/acetone) (ppm)	^{13}C-NMR ($CDCl_3$/acetone) (ppm)	^{125}Te-NMR ($CDCl_3$) (ppm)
1	1670 (asym CO) 1334 (sym CO) 505 (TeC)	2.5 (–CH_2–C) 3.2 (–CH_2–Te) 3.9 (CH_2CI)	32.3 (CCH_2) 41.7 ($TeCH_2$) 43.6 (CH_2CI) 172.3 (CO)	
2	1643 (asym CO) 1321 (sym CO) 546 (TeC)	2.6 (–CH_2–C) 3.3 (–CH_2–Te) 7.3–7.9 (–C_6H_5)	32.4 (CCH_2) 43.0 ($TeCH_2$) 128.1, 129.7, 132.1 (C_6H_5) 171.9 (CO)	640.1 (q, $^{2}J_{TeH} = 45$ Hz; $^{3}J_{TeH} = 12$ Hz)
3	1657 (asym CO) 1387 (sym CO) 525 (TeC)	2.6 (–CH_2–C) 3.4 (–CH_2–Te) 8.1–8.2 (–C_6H_4)	34.7 (CCH_2) 47.6 ($TeCH_2$) 123.4, 130.9, 137.2 (C_6H_4) 169.9 (CO)	
4	1637 (asym CO) 1341 (sym CO) 538 (TeC)	2.0 (–CH_2–C) 2.9 (–CH_2–Te) 8.9–9.0 (–C_6H_3)	30.0 (CCH_2) 32.3 ($TeCH_2$) 120.9, 122.3, 129.6 (C_6H_3) 149.1 (CO)	
5	1617 (asym CO) 1347 (sym CO) 551 (TeC)	2.5 (–CH_2–C) 3.3 (–CH_2–Te) 3.8 (–OCH3) 6.8–7.8 (–C_6H_4)	32.2 (CCH_2) 47.5 ($TeCH_2$) 43.0 (OCH3) 126.6, 131.3, 131.8 (C_6H_4) 172.5 (CO)	633.2 (q, $^{2}J_{TeH} = 45$ Hz; $^{3}J_{TeH} = 12$ Hz)

respectively. $\Delta\nu$ (ν asym COO − ν sym COO) ca. 269–335 cm^{-1} indicates the presence of unidentate carboxylate groups [6,9]. The (^{1}H, ^{13}C, ^{125}Te)-NMR data exhibit the characteristic signals at their expected positions with expected multiplicity.

*3.2. Description of the structures of **2** and **3***

Colourless crystals of **2** and pale yellow crystals of **3** were grown from acetone at room temperature.

3.2.1. Description of the structure of $C_4H_8Te(OCOC_6H_5)_2$ (2)

The structure of **2** is shown in Fig. 1. Of the four methylene groups in the heterocycle, the two methylene carbon atoms of the C_4H_8 group are bonded to the Te(IV) atom with equal Te–C bond lengths of 2.119(4) Å. The coordination geometry formed by the four closest atoms C(11), C(11)#, O(1), O(1)# is approximately trigonal bipyramidal with the oxygen atoms in the apical positions (Te–O(1) = 2.166(3), Te–O(1)# = 2.166(3) Å) and the two carbon atoms in the equatorial plane (∠C(11)#–Te–C(11), 85.5(2)°). The fifth coordination position in the equatorial plane is apparently occupied by a stereochemically active electron lone pair. An analogous coordination is shown by the Te atom in 10,10-di(trifluoroacetato)phenoxotellurine [4]. The O(1)–Te–O(1)# angle of 160.1(2)° deviates

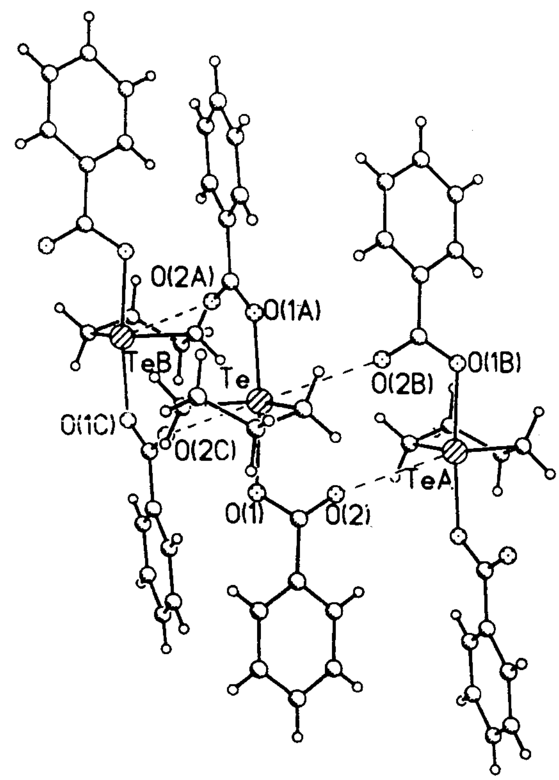

Fig. 2. Molecules of **2** connected through Te···O secondary bonds (broken).

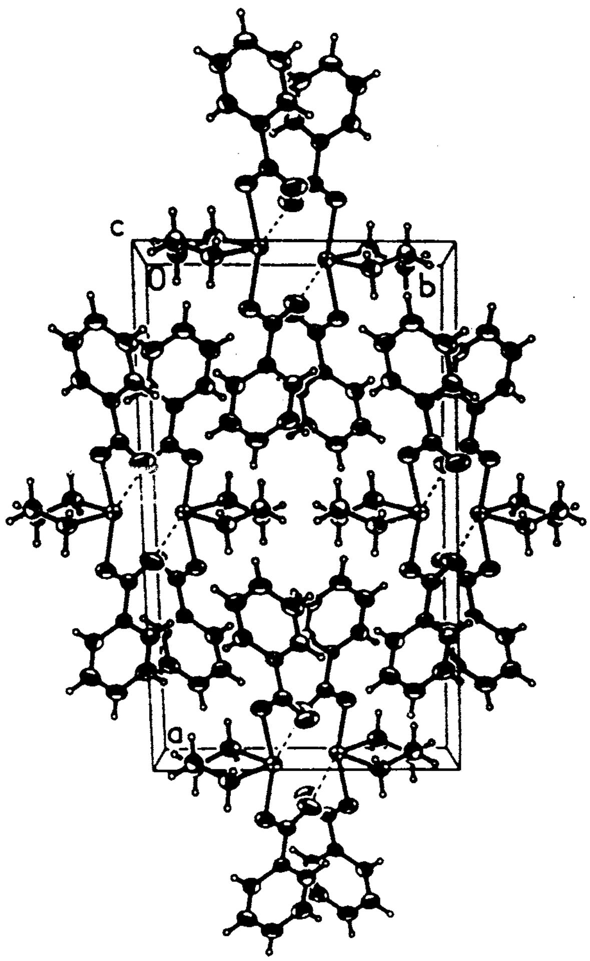

Fig. 3. The unit cell of **2** projected down *c*, showing the intermolecular Te···O secondary bonds (broken).

considerably from linearity with both oxygen atoms pushed away from the equatorial tellurium lone pair. This fairly large deviation from linearity in the O(1)–Te–O(1) # angle has also been observed in acyclic dialkyl- [6] and diaryl-tellurium dicarboxylates [10–12] and may be attributed to the presence of Te···O secondary bonds [16].

The secondary bond Te···O brings up the coordination of the Te atom to octahedral with an unshared electron pair at the vertex situated in the *trans* position to one of the $-CH_2$ groups attached to tellurium (the secondary bond itself is in the *trans* position to the other $-CH_2$ group attached to tellurium). The angles O···Te–C(11) and O···Te–C(11) # are 149.4 and 70.6° (Table 4 and Fig. 2). Similar coordination is shown by tellurium in di(trifluoroacetato)diphenyltellurium [12] and in *cis*-2-ethoxycycloheptyl tribromotellurium [13] in which the unshared electron pair is in the *trans* position to the organic group.

The Te···O distance, 3.083 Å is shorter than the sum of van der Waals radii (3.60 Å) [12,14] and longer then the sum of covalent radii (2.03 Å) [15] and thus definitely corresponds to a secondary bond [16] but it is much weaker than in the compound *cis*-2-ethoxycycloheptyl tribromotellurium [13], where the Te···O distance is 2.49 Å and in $(CH_3)_2Te(OCOC_6H_5)_2$ [6] where it is 2.224 Å, but is comparable to the Te···O distances (2.95–3.02 Å) in $(p\text{-}MeOC_6H_4)Te(OCOCH_3)_2$ [10] and (2.99–3.03 Å) in $(C_6H_5)_2Te(OCOCF_3)_2$ [12]. These weak Te···O secondary bonds join the molecules in dimeric units present in the unit cell (Fig. 3) The secondary Te···O secondary bond (O···Te–C) results in the formation of a zig–zag polymer chain (Fig. 2) in

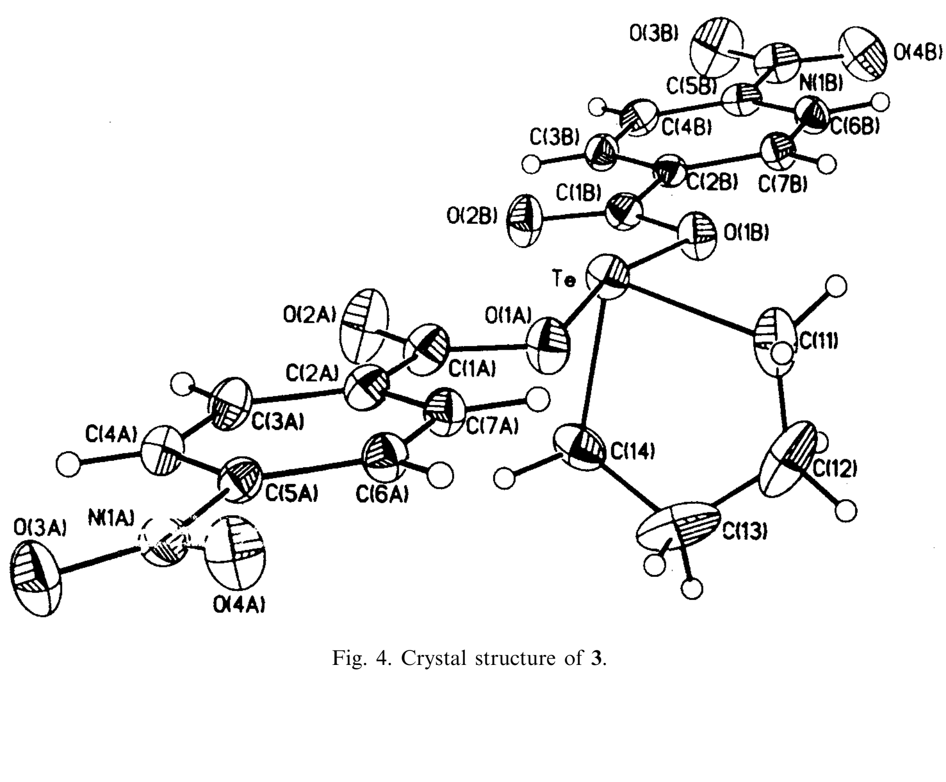

Fig. 4. Crystal structure of **3**.

which the Te atom is effectively seven coordinate, similar to the coordination shown by tellurium through the Te···S secondary bonds in S,S′-1,3-dihydro-2λ^4-benzotellurole-2,2-diyl-O,O,O′,O′-tetraethylbis(dithiophosphate) [$C_8H_8Te\{S_2P(OEt)_2\}$] [17].

3.2.2. Description of the structure of C_4H_8Te-(4-$NO_2C_6H_4OCO)_2$ (3)

The structure of **3** is shown in Fig. 4. Of the four methylene groups in the heterocycle, the two methylene carbon atoms of the C_4H_8 group are bonded to the Te(IV) atom with the Te–C(11) and Te–C(14) bond lengths 2.085(4) and 2.109(4) Å, respectively. The coordination geometry formed by the four closest atoms C(11), C(14), O(1A), O(1B) is approximately trigonal bipyramidal with the oxygen atoms in the apical positions (Te–O(1A) = 2.178(2), Te–O(1B) = 2.167(2) Å) and the two carbon atoms in the equatorial plane (∠C(11)–Te–C(14) = 86.6(2)°). The fifth coordination position in the equatorial plane is apparently occupied by a stereochemically active electron lone pair [12,13].

The O(1A)–Te–O(1B) angle of 166.54(8)° deviates considerably from linearity with both oxygen atoms pushed away from the equatorial tellurium lone pair, similar to deviations from linearity observed in acyclic dialkyl- [6] and diaryl tellurium dicarboxylates [10–12] and may be attributed to the presence of Te–O secondary bonds [16]. In the unit cell of **3** the intermolecular C–H···O interactions and Te···O secondary bonds are seen which join the molecules forming weakly bridged dimers. Refinement of the ∠CHO torsion angle is in progress to confirm whether the C–H···O interaction corresponds to the C–H···O hydrogen bond. The $-NO_2$ groups of 4-$NO_2C_6H_4OCO$ possess N(1A)–O(3A) = 1.213(4) Å, N(1A)–O(4A) = 1.219(4) Å; O(3A)–N(1A)–O(4A) = 123.1° and N(1B)–O(4B) = 1.215(5) Å, N(1B)–O(3B) = 1.213(5) Å; O(3B)–N(1B)–O(4B) = 123.8(3)°.

A comparison of the structures of **2** and **3** indicates that the former possesses a more symmetrical structure in comparison to the latter vis-à-vis oxygen atoms in apical positions to the Te and carbon atoms attached to Te in the equatorial positions.

4. Supplementary material

Crystallographic data for the structure analyses has been deposited with the Cambridge Crystallographic Data Centre, CSD-116642 for 1,1,2,3,4,5,-hexahydro-1,1-di(benzoato)tellurophene (**2**) and CSD-116643 for 1,1,2,3,4,5-hexahydro-1,1-di(4-nitrobenzoato)tellurophene (**3**). Copies of this information may be obtained free of charge from The Director, CCDC, 12, Union Road, Cambridge CB2 1EZ (fax: +44-1223-0336033 or e-mail deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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