Synthesis, spectroscopic characterisation of 1,1,2,3,4,5,6-heptahydro-1,1-di(carboxylato)telluranes and 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl dicarboxylates
Crystal structures of 1,1,2,3,4,5,6-heptahydro-1,1-di(benzoato)tellurane and 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl dibenzoate

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Abstract

1,1,2,3,4,5,6-Heptahydro-1,1-di(carboxylato)telluranes $C_5H_{10}Te(OCOR)_2$ (R = OCO, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$) and 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl dicarboxylates $C_8H_8Te(OCOR)_2$ (R = CH_3, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$, 4-$NH_2C_6H_4$) were obtained from the reactions of 1,1,2,3,4,5,6-heptahydro-1,1-diiodotellurane and 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl diiodide with silver carboxylates. They were characterised by IR and (^{1}H, ^{13}C, ^{125}Te)-NMR spectroscopy. UV, ^{13}C/^{125}Te CP/MAS spectra and thermogravimetric analysis of representative complexes are also described. The structures of $(C_5H_{10}Te(OCOC_6H_5)_2$ and $C_8H_8Te(OCOC_6H_5)_2$ were established by single crystal X-ray diffraction studies. In both cases the immediate coordination geometry about the central Te atom can be described as pseudo trigonal bipyramidal in which two oxygen atoms of the unidentate benzoate groups are in the axial positions, two methylene carbon atoms (attached to Te) of C_5H_{10} and C_8H_8 groups and the stereochemically active electron lone pair occupy equatorial positions. Te···O secondary bonds directed polymeric (*zig-zag chains*) associations are present in $C_5H_{10}Te(OCOC_6H_5)_2$ (cf. trimeric associations in the precursor $C_5H_{10}TeI_2$) and tetrameric (*stair like*) associations exist in $C_8H_8Te(OCOC_6H_5)_2$ (cf. polymeric associations in the precursor $C_8H_8TeI_2$). © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Tellurium; Carboxylates; Intermolecular secondary bonds; Supramolecular associations

1. Introduction

1,1,2,3,4,5,6-Heptahydro-1,1-diiodotellurane (C_5H_{10}-TeI_2) was synthesized by Farrar and Gulland [1]. Its derivatives have been reported from our laboratory [2] and we [3], recently, reported the X-ray structure of $C_5H_{10}TeI_2$ and supramolecular associations present in it. 1,3-Dihydro-2λ^4-benzotellurole-2,2-diyl diiodide

($C_8H_8TeI_2$) was first synthesised by Ziolo and Günther [4] by the reaction of α,α′-dichloro-*o*-xylene, Te and NaI in 2-methoxy ethanol. We [5] synthesized 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl diiodide with minor modification (α,α′-dibromo-*o*-xylene in place of α,α′-dichloro-*o*-xylene) and under easier conditions. Its structure has been determined by single crystal X-ray diffraction studies by Ziolo et al. [4,6,7] and existence of supramolecular associations in it through Te···I secondary bonds has been described by us [3]. The literature survey reveals that the reactions of 1,3-dihydro-2λ^4-benzotellurole-2,2-diyl diiodide were not investigated till 1982

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when simultaneously but independently two reports appeared; one related to the cations of 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl diiodide by McWhinnie and co-workers [8] and the other on the anions of 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl diiodide from us [5] including 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl dicarboxylates viz. $C_8H_8Te(OCOR)_2$, [R = CH_3, CH_2Cl, CF_3, C_6H_5]. These dicarboxylates were characterised by IR and ^{1}H-NMR spectroscopy. Later the single crystal X-ray diffraction study of $C_8H_8Te(OCOCH_3)_2$, was reported by Dakternieks and co-workers [9].

In view of no structural study reported for 1,1,2,3,4,5,6-heptahydro-1,1-di(carboxylato)telluranes and in continuation of our structural studies on hypervalent Te(IV) compounds [3,10–13], in the present investigation, we describe a comprehensive study on synthesis and characterisation of 1,1,2,3,4,5,6-heptahydro-1,1-dicarboxylato telluranes $C_5H_{10}Te(OCOR)_2$ [R = OCO, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$] and 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl dicarboxylates $C_8H_8Te(OCOR)_2$, [R = CH_3, C_6H_5, 4-$NO_2C_6H_4$, 3,5-$(NO_2)_2C_6H_3$, 4-$OCH_3C_6H_4$, 4-$NH_2C_6H_4$]; X-ray structures of $C_5H_{10}Te(OCOC_6H_5)_2$ (**2**) and $C_8H_8Te(OCOC_6H_5)_2$ (**7**) with reference to supramolecular associations and thermal effects.

Supramolecular chemistry of organometallic compounds is an area of current interest [3,13,14] because of their usefulness in materials' chemistry [14e,14h]. Further thermal effects on organoselenium compounds have been examined [15] whereas, to our knowledge, there is no report of thermal effects on organotelluriums.

2. Experimental

2.1. Physical measurement

Elemental analyses for C, H, and N were carried out on an Elemental Analyser Heraeus Carlo Erba 1108 and Te was estimated volumetrically [5]. 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 (^{1}H, ^{13}C, ^{125}Te)-NMR spectra were recorded on a Varian VXR3005 spectrometer in CDCl$_3$, for all the complexes except those of 1,1,2,3,4,5,6-heptahydro-1,1-di(3,5-dinitrobenzoato) tellurane (**4**) and 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl di(3,5-dinitrobenzoate) (**9**) which were recorded in Me$_2$SO and C$_3$H$_6$O, respectively. The operating frequency for ^{125}Te-NMR was 94.752 MHz with a pulse width of 42.8°. The 50.3 MHz ^{13}C and 63.2 MHz ^{125}Te CP/MAS solid state NMR spectral studies were carried out at 3.5 and 4.7 KHz at Institut für Anorganische Chemie Universität des Saarlandes, Saarbrücken, Germany. ^{125}Te-NMR spectra were referenced to Me$_2$Te ($\delta = 0$ ppm). The

UV spectral data of the solutions of the complexes in MeOH were recorded on an Unicam SP 500 spectrometer with 10 mm path cell. The thermogravimetric analyses were carried out on a Thermogravimetric analyses apparatus fabricated by Fertiliser Corporation of India Sindri (Bihar). The single crystal X-ray diffraction studies were carried out at the Chemistry Department, Howard University, Washington DC.

2.2. Synthesis

1,1,2,3,4,5,6-Heptahydro-1,1-diiodotellurane (C_5H_{10}-TeI$_2$) [1] and 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl diiodide ($C_8H_8TeI_2$) [5] were prepared by the literature methods. $C_5H_{10}TeI_2$ was recrystallised from C_3H_6O to obtain orange–brown needles and $C_8H_8TeI_2$ was recrystallised from hot 2-methoxy ethanol to obtain yellow–orange crystals.

1,1,2,3,4,5,6-Heptahydro-1,1-mono(oxalato)tellurane (**1**), 1,1,2,3,4,5,6-heptahydro-1,1-di(benzoato)tellurane (**2**), 1,1,2,3,4,5,6-heptahydro-1,1-di(4-nitrobenzoato)tellurane (**3**), 1,1,2,3,4,5,6-heptahydro-1,1-di(3,5-dinitrobenzoato)tellurane (**4**), and 1,1,2,3,4,5,6 heptahydro-1,1-di(4-methoxybenzoato)tellurane (**5**); 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl diacetate (**6**); -dibenzoate (**7**); -di(4-nitrobenzoate) (**8**); -di(3,5-dinitrobenzoate) (**9**);-di(4-methoxybenzoate) (**10**); -di(4-aminobenzoate) (**11**) were prepared by stirring $C_5H_{10}TeI_2$ or $C_8H_8TeI_2$ with freshly prepared silver carboxylates in C_3H_6O or CH_2Cl_2.
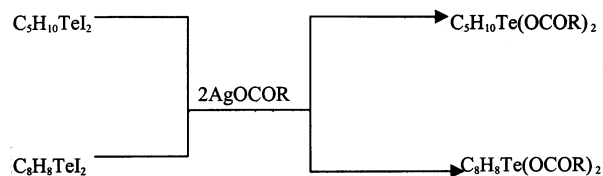
(R=OCO,CH$_3$,C$_6$H$_5$, 4-NO$_2$C$_6$H$_4$, 3,5-(NO$_2$)$_2$C$_6$H$_3$, 4-OCH$_3$C$_6$H$_4$, 4-NH$_2$C$_6$H$_4$)

To 1,2,3,4,5,6-heptahydro-1,1-diiodotellurane(2 g, 4.42 mmol) was added freshly prepared silver oxalate (1.73 g, 8.85 mmol), silver benzoate (2.03 g, 8.86 mmol), silver 4-nitro benzoate (2.42 g, 8.85 mmol), silver 3,5-dinitro benzoate (2.82 g, 8.86mo1), silver 4-methoxy benzoate (2.29 g, 8.85 mmol) in C_3H_6O (approximately 30 ml). It was stirred for 4 h and filtered to eliminate AgI and excess silver carboxylate. The filtrate was concentrated under reduced pressure to give complexes **1–5**. Complex **1** yield: 0.85 g (67%), m.p. 120 °C. Anal. Calc. for $C_7H_{10}O_4Te$: C, 29.41; H, 3.50; Te, 44.67. Found: C, 29.35; H, 3.45; Te, 44.62%. Complex **2** yield: 1.20 g (62%), m.p. 138–140 °C. Anal. Calc. for $C_{19}H_{20}O_4Te$: C, 51.86; H, 4.54; Te, 29.02. Found: C, 51.72; H, 4.45; Te, 29.00%. Complex **3** yield: 1.46 g (62%), m.p. 128–130 °C. Anal. Calc. for $C_{19}H_{18}O_8N_2Te$: C, 43.05; H, 3.39; N, 5.28; Te, 24.09. Found: C, 43.02; H, 3.32; N,

5.15; Te, 24.05%. Complex **4** yield: 1.65 g (60%), m.p. 225–230 °C. Anal. Calc. for $C_{19}H_{16}O_{12}N_4Te$: C, 36.79; H, 2.58; N, 9.03; Te, 20.59. Found: C, 36.72; H, 2.54; N, 9.00; Te, 20.56%. Complex **5** yield: 1.50 g (68%), m.p. 150 °C. Anal. Calc. for $C_{21}H_{24}O_6Te$: C, 50.44; H, 4.80; Te, 25.54. Found: C, 50.42; H, 4.75; Te, 25.48%.

To 1,3-dihydro-$2\lambda^4$-benzotellurole-2,2-diyl diiodide (2 g, 4.12 mmol) was added freshly prepared silver acetate (1.38 g, 8.25 mmol), silver benzoate (1.89 g, 8.25 mmol), silver 4-nitro benzoate (2.26 g, 8.25 mmol), silver 3,5-dinitro benzoate (2.63 g, 8.25 mmol), silver 4-methoxy benzoate (2.14 g, 8.25 mmol), silver 4-amino benzoate (2.01 g, 8.25 mmol) in CH_2Cl_2 (approximately 30 ml). It was stirred for 6 h and filtered to eliminate AgI and excess silver carboxylate. The filtrate was concentrated under reduced pressure to give complexes **6**–**11**. Complex **6** yield: 0.34 g (24%), m.p. 150 °C. Anal. Calc. for $C_{12}H_{14}O_4Te$: C, 41.18; H, 4.00; Te, 36.49. Found: C, 41.10; H, 4.01; Te, 36.46%. Complex **7** yield: 0.42 g (22%), m.p. 150 °C. Anal. Calc. for $C_{22}H_{18}O_4Te$: C, 55.74; H, 3.80; Te, 26.94. Found: C, 55.72; H, 3.78; Te, 26.88%. Complex **8** yield: 0.29 g (13%), m.p. 148–150 °C. Anal. Calc. for $C_{22}H_{16}O_8N_2Te$: C, 46.84; H, 2.83; N, 4.96; Te, 22.64. Found: C, 46.80; H, 2.77; N, 4.94; Te, 22.60%. Complex **9** yield: 0.90 g (33%), m.p. 118–120 °C. Anal. Calc. for $C_{22}H_{14}O_{12}N_4Te$: C, 40.39; H, 2.14; N, 8.56; Te, 19.52. Found: C, 40.35; H, 2.12; N, 8.54; Te, 19.50%. Complex **10** yield: 0.63 g (29%), m.p. 160–162 °C. Anal. Calc. for $C_{24}H_{22}O_6Te$: C, 53.97; H, 4.12; Te, 23.91. Found: C, 53.91; H, 4.11; Te, 23.86%. Complex **11** yield: 0.21 g (13%), m.p. 120–125 °C. Anal. Calc. for $C_{22}H_{20}O_4N_2Te$: C, 70.21; H, 5.31; N, 7.44; Te, 33.93. Found: C, 70.11; H, 5.25; N, 7.42; Te, 33.91%.

2.3. X-ray measurements

Colourless needle shaped crystals $(0.25 \times 0.4 \times 0.4$ mm) of complex **2** at [158(2) K], light brown prism shaped crystals $(0.35 \times 0.67 \times 0.55$ mm) of complex **2** at [293(2) K] and colourless crystals $(0.20 \times 0.28 \times 0.34$ mm) of complex **7** at 293(2) K were mounted on a Bruker P4S diffractometer using graphite monochromatic Mo–K_α radiation (0.71073 Å). The unit cells were determined from 25 randomly selected reflections using the automatic search index and least-squares refinement. The structures of **2** and **7** were solved in space group $P2_1/c$. The data were corrected for Lorentz, polarization and absorption effects. The data were monitored by measuring three standard reflections at every 97 reflections. The structures were solved by the routine heavy atom method SHELXS-86 [16] and Fourier methods and refined by full-matrix least-squares using the SHELXL-93 program [17] with the non-hydrogen atoms anisotropic and hydrogen atoms having fixed isotropic thermal parameters of 0.08 Å^2 (Table 1).

Selected bond lengths and bond angles for **2** and **7** are listed in Tables 2–5. The crystal structures and supramolecular associations of **2** and **7** are shown in Figs. 1–2 and 3–4, respectively.

3. Results and discussion

3.1. Spectroscopic characterization

Spectroscopic data for $(C_5H_{10}Te(OCOR)_2)$ **1**–**5** and $(C_8H_8Te(OCOR)_2)$ **6**–**11** are given in Tables 6 and 7 ,respectively. The IR spectra of **1**–**11** display the characteristic carboxylate group frequencies. The v asym COO and v sym COO are in the range 1603–1650 and 1292–1394 cm^{-1}, respectively. Δv (v asym COO–v sym COO) approximately 278–353 cm^{-1} indicate the presence of unidentate carboxylate groups in all the complexes [11,12,18] except in the case of **1** ($\Delta v = 243$ cm^{-1}) which appears to contain bidentate oxalate group [11,19]. The solution (^{1}H, ^{13}C, ^{125}Te)-NMR data exhibit the characteristic signals at their expected positions with expected multiplicity. The ^{13}C CP/MAS solid state NMR of **2** shows CH$_2$–Te (32 ppm), CH$_2$–CH$_2$ (30, 26 ppm), C$_6$H$_5$ (134, 129, 128 ppm), CO (172 ppm) [cf. CH$_2$–Te (29.8 ppm), CH$_2$–CH$_2$ (27.1, 20.4 ppm), C$_6$H$_5$ (132.2, 129.8, 128.1 ppm), CO (171.8 ppm) in solution ^{13}C-NMR (Table 6)] and ^{125}Te CP/MAS NMR of **2** shows isotropic shift at 902 ppm which is comparable to that of solution ^{125}Te-NMR ($\delta = 918$ ppm).

The ultra violet spectra of $C_8H_8TeI_2$ and $C_8H_8Te(OCOC_6H_5)_2$ in conjunction with their X-ray structures in terms of supramolecular associations are described as follows:

Two maxima in the spectrum of $C_8H_8TeI_2$ are observed. The maxima at 271 nm is attributed to (π, π^*) benzoid transition in accordance with the earlier observations [4,20]. The absorption at 336 nm appears to be because of the transfer of electron density from the iodine into the d-orbitals of Te termed as *charge-transfer transition* [21,22] presumably, due to Te···I secondary bonds in the polymeric associations [3] existing in $C_8H_8TeI_2$.

In $C_8H_8Te(OCOC_6H_5)_2$ (**7**), the absorption located at 272 nm is attributed to (π, π^*) benzoid transition as in the case of $C_8H_8TeI_2$. The absorption at 280 nm is apparently because of the transfer of electron density from $>C=O$ into the d-orbital of Te termed as *charge-transfer transition* [21,22], presumably, due to Te···O secondary bonds in the tetrameric supramolecular associations (present work) existing in $C_8H_8Te(OCOC_6H_5)_2$.
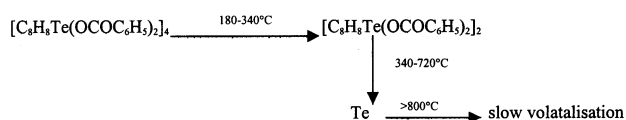
Table 1
Crystal data and refinement details for complexes **2** and **7**

	2 (colourless needle)	**2** (brown prism)	**7**
Empirical formula	$C_{19}H_{20}O_4Te$	$C_{19}H_{20}O_4Te$	$C_{22}H_{18}O_4Te$
Formula weight	439.95	439.95	473.96
Temperature (K)	158(2)	293(2)	293(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
a (Å)	10.0876(8)	10.2162(8)	19.3018(14)
b (Å)	17.0245(12)	17.1596(17)	9.2682(7)
c (Å)	10.6499(8)	10.7094(10)	10.8086(8)
α (°)	90	90	90
β (°)	108.8100(10)	109.551(6)	93.5190(10)
γ (°)	90	90	90
V (Å^3)	1731.3(2)	1769.2(3)	1929.9(2)
Z	4	4	4
μ (mm^{-1})	1.738	1.700	1.566
Index ranges	$-12 \leq h \leq 12$, $-7 \leq k \leq 21$, $-12 \leq l \leq 13$	$0 \leq h \leq 13$, $-22 \leq k \leq 0$, $-13 \leq l \leq 13$	$-25 \leq h \leq 25$, $-12 \leq k \leq 11$, $-13 \leq l \leq 14$
Reflections collected	11 788	4289	20 774
Independent reflections	3377 ($R_{int} = 0.0297$)	4071 ($R_{int} = 0.0164$)	4696 ($R_{int} = 0.0539$)
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0186$, $wR_2 = 0.0487$	$R_1 = 0.0294$, $wR_2 = 0.0710$	$R_1 = 0.0262$, $wR_2 = 0.0623$
R indices (all data)	$R_1 = 0.0211$, $wR_2 = 0.0496$	$R_1 = 0.0409$, $wR_2 = 0.0755$	$R_1 = 0.0380$, $wR_2 = 0.0660$

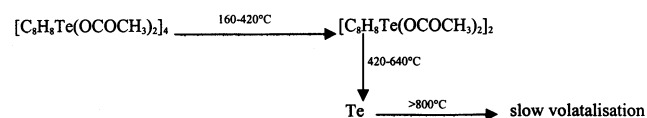## 3.2. Thermogravimetric analysis

With a view to examine the thermal stability and to obtain monomer of the complexes, thermogravimetric analysis of two representative complexes viz. $C_8H_8Te(OCOC_6H_5)_2$ (**7**) and $C_8H_8Te(OCOCH_3)_2$ (**6**) were carried out at a heating rate of 5 °C min^{-1}. The pyrolysis curves are produced in Fig. 5.

Thermogravimetric data coupled with single crystal X-ray diffraction data of $C_8H_8Te(OCOC_6H_5)_2$, (**7**) indicate that there is no appreciable weight loss below 180 °C. After 180 °C the decomposition of the tetrameric unit of the complex starts and at 340 °C the mass corresponds to dimeric unit of the complex. The observed percentage weight loss is 51 in agreement with the theoretical value of 50. The dimeric unit in the temperature range 340–720 °C undergoes decomposition to yield tellurium metal (Found% weight loss 73, Calc.% weight loss 73). Above 800 °C the mass corresponding to tellurium metal decreases which is attributed to slow volatilisation of the metal (m.p. 450 °C, b.p. 990 °C). The scheme of decomposition may be represented as follows:

$$[C_8H_8Te(OCOC_6H_5)_2]_4 \xrightarrow{180-340°C} [C_8H_8Te(OCOC_6H_5)_2]_2$$
$$\downarrow 340-720°C$$
$$Te \xrightarrow{>800°C} \text{slow volatalisation}$$

Almost the same pattern is observed for $C_8H_8Te(OCOCH_3)_2$ (**6**), where there is no appreciable weight loss below 160 °C. After 160 °C, the complex starts decomposing and at 420 °C the mass corresponds to

dimeric unit of the complex. The observed percentage weight loss is 51 in agreement with the theoretical value of 50. The dimeric unit in the temperature range 420–640 °C undergoes decomposition to yield tellurium metal (Found% weight loss 64, Calc.% weight loss 64). The metal slowly volatilises above 800 °C.

$$[C_8H_8Te(OCOCH_3)_2]_4 \xrightarrow{160-420°C} [C_8H_8Te(OCOCH_3)_2]_2$$
$$\downarrow 420-640°C$$
$$Te \xrightarrow{>800°C} \text{slow volatalisation}$$

3.3. Description of the structures of 2 and 7

Colourless needle shaped crystals of **2**, light brown prism shaped crystals of **2** were grown from benzene and acetone, respectively and colourless crystals of **7** were grown from acetone at room temperature.

3.3.1. Description of the structure of $C_5H_{10}Te(OCOC_6H_5)_2$ (2)

The structure of **2** is shown in Fig. 1. In $C_5H_{10}Te(OCOC_6H_5)_2$ the tellurium atom completes a cyclohexane ring with other five carbon atoms in $C_5H_{10}Te$ cycle. The average C–C–C angles are 114°. The three carbon atoms at the other end of cyclohexane ring make an angle $\angle C(2)-C(3)-C(4) = 112.96(2)°$. The $C_5H_{10}Te$ ring has chair conformation in which part of the ring is flattened and a part is considerably puckered. A similar conformation for $C_5H_{10}Te$ cycle has been envisaged by us [3] in 1,2,3,4,5,6-heptahydro-1,1-diiodo tellurane (X-ray evidence) and in 1,2,3,4,5,6-heptahy-

Table 2
Bond lengths (Å) and angles (°) for **2**

Bond lengths

Te−C(1)	2.127(2)	Te−C(5)	2.132(2)
Te−O(1B)	2.157(1)	Te−O(1A)	2.167(1)
O(1A)−C(1A)	1.306(2)	O(2A)−C(1A)	1.224(2)
O(1B)−C(1B)	1.312(2)	O(2B)−C(1B)	1.222(3)
C(1)−C(2)	1.520(3)	C(2)−C(3)	1.531(3)
C(3)−C(4)	1.526(3)	C(4)−C(5)	1.525(3)
C(1A)−C(2A)	1.501(3)	C(2A)−C(3A)	1.390(3)
C(2A)−C(7A)	1.398(3)	C(3A)−C(4A)	1.380(3)
C(4A)−C(5A)	1.383(3)	C(5A)−C(6A)	1.388(3)
C(6A)−C(7A)	1.390(3)	C(1B)−C(2B)	1.498(3)
C(2B)−C(7B)	1.390(3)	C(2B)−C(3B)	1.395(3)
C(3B)−C(4B)	1.388(3)	C(4B)−C(5B)	1.377(4)
C(5B)−C(6B)	1.385(4)	C(6B)−C(7B)	1.389(3)

Bond angles

C(1)−Te−C(5)	97.89(7)	C(1)−Te−O(1B)	83.80(7)
C(5)−Te−O(1B)	85.97(6)	C(1)−Te−O(1A)	86.27(7)
C(5)−Te−O(1A)	82.51(6)	O(1B)−Te−O(1A)	163.60(5)
C(1A)−O(1A)−Te	113.71(12)	C(1B)−O(1B)−Te	109.89(12)
C(2)−C(1)−Te	115.52(13)	C(1)−C(2)−C(3)	115.45(17)
C(4)−C(3)−C(2)	112.96(16)	C(5)−C(4)−C(3)	114.27(16)
C(4)−C(5)−Te	113.64(12)	O(2A)−C(1A)−O(1A)	123.1(2)
O(2A)−C(1A)−C(2A)	122.11(19)	O(1A)−C(1A)−C(2A)	114.81(16)
C(3A)−C(2A)−C(7A)	119.67(19)	C(3A)−C(2A)−C(1A)	119.19(18)
C(7A)−C(2A)−C(1A)	121.12(17)	C(4A)−C(3A)−C(2A)	120.4(2)
C(3A)−C(4A)−C(5A)	120.1(2)	C(4A)−C(5A)−C(6A)	120.1(2)
C(5A)−C(6A)−C(7A)	120.1(2)	C(6A)−C(7A)−C(2A)	119.55(18)
O(2B)−C(1B)−O(1B)	123.19(18)	O(2B)−C(1B)−C(2B)	121.46(18)
O(1B)−C(1B)−C(2B)	115.34(17)	C(7B)−C(2B)−C(3B)	120.24(19)
C(7B)−C(2B)−C(1B)	121.51(18)	C(3B)−C(2B)−C(1B)	118.22(19)
C(4B)−C(3B)−C(2B)	119.7(2)	C(5B)−C(4B)−C(3B)	119.7(2)
C(4B)−C(5B)−C(6B)	121.0(2)	C(5B)−C(6B)−C(7B)	119.7(2)
C(6B)−C(7B)−C(2B)	119.6(2)		

Symmetry transformations used to generate equivalent atoms.

Table 3
Bond lengths (Å) and angles (°) for **2**

TE	O1A	2.167				
TE	O1B	2.157	163.6			
TE	C1	2.127	86.3	83.8		
TE	C5	2.132	82.5	86.0	97.9	
TIE	O2AA	3.307	118.0	70.9	71.6	155.2
			O1A	O1B	C1	C5
O2A	C1A	1.224				
O2A	TEA	3.307	169.2			
			C1A			

Table 4
Bond lengths (Å) and angles (°) for **7**

Bond lengths

Te−C(8)	2.106(2)	Te−C(1)	2.111(2)
Te−O(1B)	2.141(2)	Te−O(1A)	2.143(2)
O(1A)−C(7A)	1.313(3)	O(2A)−C(7A)	1.217(3)
O(1B)−C(7B)	1.323(3)	O(2B)−C(7B)	1.219(3)
C(1A)−C(2A)	1.384(3)	C(1A)−C(6A)	1.398(3)
C(1A)−C(7A)	1.490(31	C(2A)−C(3A)	1.388(4)
C(3A)−C(4A)	1.364(4)	C(4A)−C(5A)	1.375(4)
C(5A)−C(6A)	1.374(3)	C(1B)−C(2B)	1.379(3)
C(1B)−C(6B)	1.389(4)	C(1B)−C(7B)	1.483(3)
C(2B)−C(3B)	1.376(4)	C(3B)−C(4B)	1.356(4)
C(4B)−C(5B)	1.366(4)	C(5B)−C(6B)	1.380(4)
C(1)−C(2)	1.503(3)	C(2)−C(3)	1.392(3)
C(2)−C(7)	1.395(3)	C(3)−C(4)	1.376(4)
C(4)−C(5)	1.381(4)	C(5)−C(6)	1.379(3)
C(6)−C(7)	1.396(3)	C(7)−C(8)	1.509(3)

Bond angles

C(8)−Te−C(1)	86.89(8)	C(8)−Te−O(1B)	80.86(7)
C(1)−Te−O(1B)	85.94(7)	C(8)−Te−O(1A)	80.82(7)
C(1)−Te−O(1A)	87.80(8)	O(1B)−Te−O(1A)	160.92(7)
C(7A)−O(1A)−Te	118.49(16)	C(7B)−O(1B)−Te	112.46(14)
C(2A)−C(1A)−C(6A)	119.2(2)	C(2A)−C(1A)−C(7A)	119.6(2)
C(6A)−C(1A)−C(7A)	121.2(2)	C(1A)−C(2A)−C(3A)	119.7(3)
C(4A)−C(3A)−C(2A)	120.6(3)	C(3A)−C(4A)−C(5A)	120.2(3)
C(6A)−C(5A)−C(4A)	120.3(3)	C(5A)−C(6A)−C(1A)	120.1(2)
O(2A)−C(7A)−O(1A)	122.9(2)	O(2A)−C(7A)−C(1A)	123.0(2)
O(1A)−C(7A)−C(1A)	114.0(2)	C(2B)−C(1B)−C(6B)	118.2(2)
C(2B)−C(1B)−C(7B)	119.7(2)	C(6B)−C(1B)−C(7B)	122.2(2)
C(3B)−C(2B)−C(1B)	120.0(3)	C(4B)−C(3B)−C(2B)	120.4(3)
C(3B)−C(4B)−C(5B)	120.0(3)	C(4B)−C(5B)−C(6B)	120.4(3)
C(5B)−C(6B)−C(1B)	120.2(3)	O(2B)−C(7B)−O(1B)	121.9(2)
O(2B)−C(7B)−C(1B)	122.9(2)	O(1B)−C(7B)−C(1B)	115.1(2)
C(2)−C(1)−Te	106.53(13)	C(3)−C(2)−C(7)	119.8(2)
C(3)−C(2)−C(1)	120.30(18)	C(7)−C(2)−C(1)	119.88(17)
C(4)−C(3)−C(2)	120.3(2)	C(3)−C(4)−C(5)	120.2(2)
C(6)−C(5)−C(4)	120.0(2)	C(5)−C(6)−C(7)	120.7(2)
C(2)−C(7)−C(6)	110.96(18)	C(2)−C(7)−C(8)	120.04(18)
C(6)−C(7)−C(8)	120.99(18)	C(7)−C(8)−Te	106.31(14)

Symmetry transformation used to generate equivalent atoms.

Table 5
Bond lengths (Å) and angles (°) for **7**

TEA	O1AA	2.143				
TEA	O1BB	2.141	160.9			
TEA	C1AA	2.111	87.8	85.9		
TEA	C8AA	2.106	80.8	80.8	86.8	
TEA	O2A	3.376	94.8	85.4	160.6	74.6
			O1A	O1B	C1	C8

dro-1,1-dibromo tellurane by Lambert and Keske [23] (NMR evidence).

Of the five methylene groups, the two methylene carbon atoms of the C_5H_{10} group are bonded to Te(IV) atom with almost equal Te−C(1) = 2.127(2) and Te−C(5) = 2.132(2) bond lengths. The coordination geometry formed by the four closest atoms C(1), C(5), O(1A), O(1B) is approximately trigonal bipyramidal with the oxygen atoms of the unidentate benzoate groups in the

apical positions (Te−O(1A) = 2.167(1), Te−O(1B) = 2.157(1) Å) and the two methylene carbon atoms in the equatorial plane (∠ C(1)−Te−C(5), 97.89(7)°). 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(trifluoro acetato) phenoxotellurine [24], 1,1,2,3,4,5-hexahydro-1,1-di(benzoato)- and
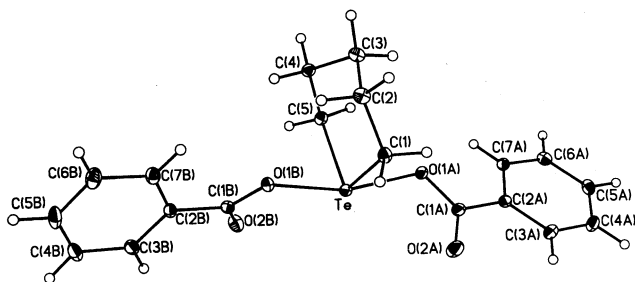
Fig. 1. Crystal structure of **2**.

1,1-di(4-nitrobenzoato)tellurophene [12]. The O(1B)Te–O(1A) angle of 163.60(5)° deviates considerably from linearity with both oxygen atoms pushed away from the equatorial tellurium lone pair. This fairly large deviation from linearity has also been observed in acyclic dialkyl-[11], diaryl- [25–27] and heterocyclic tellurium dicarboxylates [12] and may be attributed to the presence of Te···O secondary bonds [28]. The Te···O secondary bond 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 other CH_2 group attached to tellurium). The angles O···Te–C(1) and O···Te–C(1A) are 71.6 and 169.2° (Fig. 2), (Table 3). Similar coordination

is shown by tellurium in 1,1,2,3,4,5-hexahydro-1,1-di(benzoato) tellurophene [12], di(trifluoro acetate) diphenyl tellurium [27] and in *cis*-2-ethoxycycloheptyl tribromotellurium [29] in which the unshared electron pair is in the *trans* position to the organic group.

The Te···O distance 3.307 Å is shorter than the sum of van der Waals radii (3.60 Å) [27,30] and longer than the sum of covalent radii (2.03 Å) [31] and thus definitely corresponds to a secondary bond [28] but it is much weaker than in the complex *cis*-2-ethoxycycloheptyl tribromotellurium [29] where the Te···O distance is 2.49 Å and in $(CH_3)_2Te(OCOC_6H_5)_2$ [11] where it is 2.224 Å, but is comparable to the Te···O distances (2.95–3.02 Å) in $(p\text{-}MeOC_6H_4)_2Te(OCOCH_3)_2$ [25], (2.99–3.03 Å) in $(C_6H_5)_2Te(OCOCF_3)_2$ [27] and 3.083 Å in $C_4H_8Te(OCOC_6H_5)_2$ [12]. These weak Te···O secondary bonds join the molecules to form dimeric units (Fig. 2) which join together resulting in the formation of polymeric (*zig-zag chains*) supramolecular associations in which Te atom is effectively seven-coordinate, similar to the coordination shown by tellurium through Te···O secondary bonds in 1,1,2,3,4,5-hexahydro-1,1-di(benzoato)tellurophene [12] and through Te···S secondary bonds in *S,S′*-1,3-dihydro-2λ⁴-benzotellurole-2,2-diyl-*O,O,O′,O′*-tetraethyl bis-(dithiophosphate) $[C_8H_8Te\{S_2P(OEt)_2\}]$ [32] whereas in the precursor $(C_5H_{10}TeI_2)$ trimeric associations [3] are present.

Table 6
Spectroscopic data for **1**–**5**

Complex	IR (KBr) (cm^{-1})	^{1}H-NMR (CDCl$_3$) (ppm)	^{13}C-NMR (CDCl$_3$) (ppm)	^{125}Te-NMR (CDCl$_3$) (ppm)
1	1637 (asym CO) 1394 (sym CO) 505 (TeC)	1.9, 2.3 ($-CH_2-C$) 3.6 ($-CH_2-Te$)	28.8, 37.5 (CCH_2) 49.8 (TeCH_2) 158.2 (CO)	
2	1614 (asym CO) 1298 (sym CO) 505 (TeC)	1.8, 2.0 ($-CH_2-C$) 3.3 ($-CH_2-Te$) 7.4, 8.0 (C_6H_5)	20.4, 27.1 (CCH_2) 29.8 (TeCH_2) 128.1, 129.8 132.2 (C_6H_5) 171.8 (CO)	918 (q, $^2J_{TeH}$ = 43 Hz $^3J_{TeH}$ = 11 Hz)
3	1639 (asym CO) 1315 (sym CO) 518 (TeC)	1.8, 2.0 ($-CH_2-C$) 3.6 ($-CH_2-Te$) 7.8, 8.2 (C_6H_4)	35.0 (CCH_2) 45.1 (TeCH_2) 128.5, 136.0 144.0 (C_6H_4) 155.0 (CO)	
4	1622 (asym CO) 1344 (sym CO) 530 (TeC)	1.9, 3.0 ($-CH_2-C$) 3.6 ($-CH_2-Te$) 8.9, 9.0 (C_6H_{13})	18.8, 29.2 (CCH_2) 38.5 (TeCH_2) 120.1, 127.9 138.3 (C_6H_3) 165.4 (CO)	
5	1650 (asym CO) 1326 (sym CO) 510 (TeC)	1.8, 2.0 ($-CH_2-C$) 3.2 ($-CH_2-Te$) 3.8 ($-OCH_3$) 6.9, 8.1(C_6H_4)	20.8, 27.5 (CCH_2) 30.2 (TeCH_2) 55.8 (OCH_3) 113.8, 124.8, 132.1 (C_6H_4) 172.0 (CO)	

Table 7
Spectroscopic data for **6–11**

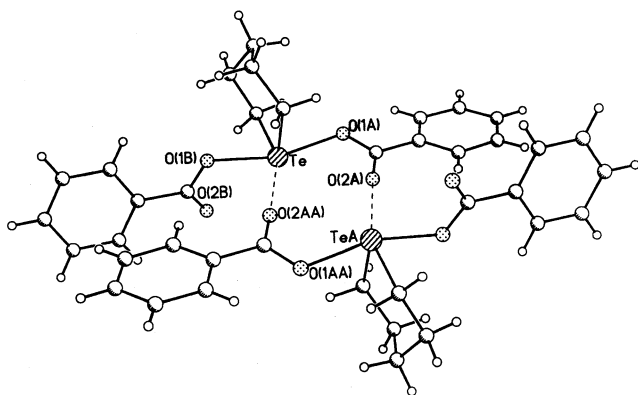| Complex	IR (KBr) (cm^{-1})	^{1}H-NMR (CDCl$_3$) (ppm)	^{13}C-NMR (CDCl$_3$) (ppm)	^{125}Te-NMR (CDCl$_3$) (ppm)
6	1645 (asym CO)	4.47 (–CH$_2$–Te)	22.1 (CH$_3$)	
	1292 (sym CO)	7.25 (C$_6$H$_4$)	46.3 (TeCH$_2$)	1070
	502 (TeC)	1.90 (CH$_3$)	128.1, 130.5,	(q, $^2J_{TeH}$ = 42 Hz)
			139.4 (C$_6$H$_4$)	
			178.5 (CO)	
7	1624 (asym CO)	4.73 (–CH$_2$–Te)	46.8 (TeCH$_2$)	
	1323 (sym CO)	7.34 (C$_6$H$_4$)	128.1, 128.4	1078
	555 (TeC)	7.88 (C$_6$H$_5$)	130.4 (C$_6$H$_4$)	(q, $^2J_{TeH}$ = 42 Hz)
			131.9, 132.7	
			139.5 (C$_6$H$_5$)	
			173.1 (CO)	
8	1633 (asym CO)	4.78 (–CH$_2$–Te)	46.8 (TeCH$_2$)	
	1321 (sym CO)	7.31 (C$_6$H$_4$)	127.5, 128.0	1103
	530 (TeC)	8.07,	129.0 (C$_6$H$_4$)	(q; $2J_{TeH}$ = 43 Hz)
		8.21(C$_6$H$_5$)	130.0, 130.2	
			136.0 (C$_6$H$_4$)	
			171.0 (CO)	
9	1628 (asym CO)	4.92 (–CH$_2$–Te)		
	1346 (sym CO)	7.36 (C$_6$H$_4$)		
	540 (TeC)	8.90, 8.99 (C$_6$H$_3$)		
10	1612 (asym CO)	4.68 (–CH$_2$–Te)	30.1 (OCH$_3$)	
	1325 (sym CO)	7.26 (C$_6$H$_4$)	46.7 (TeCH$_2$)	1064
	549 (TeC)	3.81 (OCH$_3$)	124.3, 128.0,	(q, $^2J_{TeH}$ = 41 Hz)
		6.81,	130.4 (C$_6$H$_4$)	
		7.84 (C$_6$H$_4$)	132.2, 139.6 (C$_6$H$_4$)	
			172.9 (CO)	
11	1603 (asym CO)	4.76 (–CH$_2$–Te)		
	1321 (sym CO)	7.26 (C$_6$H$_4$)		1064
	530 (TeC)	6.58, 7.73 (C$_6$H$_4$)		(q, $^2J_{TeH}$ = 38 Hz)

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

Although by using different solvents for crystallization, it was possible to isolate two type of crystals colourless needles (m.p. 138–140 °C) from benzene and light brown prisms (m.p. 148–150 °C) from acetone and initially we speculated them to be polymorphs but on the basis of X-ray diffraction data (Table 1), we conclude that they are not polymorphs.

3.3.2. Description of the structure of C$_8$H$_8$Te(OCOC$_6$H$_5$)$_2$ (7)

The structure of **7** is shown in Fig. 3. The two methylene carbon atoms of the C$_8$H$_8$ group are bonded to the Te(IV) atom with almost equal Te–C bond lengths Te–C(l) = 2.111(2) and Te–C(8) = 2.106(2) Å. The coordination geometry formed by the four closest atoms C(l), C(8), O(1A), O(1B) is approximately trigonal bipyramidal with the oxygen atoms in the apical positions [Te–O(1A) = 2.143(2), Te–O(1B) = 2.141(2) Å] and the two carbon atoms in the equatorial plane
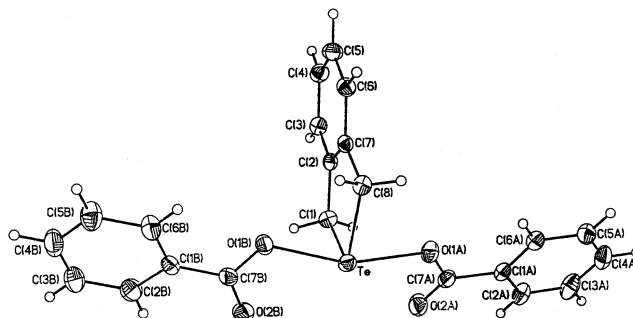

Fig. 3. Crystal structure of **7**.

[C(1)–Te–C(8) = 86.89(8)°]. The fifth coordination position, in the equatorial plane, is apparently occupied by a stereochemically active lone pair of electrons. The O(1A)–Te–O(1B) angle of 160.92(7)° deviates considerably from linearity with both oxygen atoms pushed away from the equatorial tellurium lone pair. This pseudotrigonal bipyramidal arrangement around tellurium and fairly large deviation of $\angle$ O–Te–O from linearity is in accordance with the literature reports as quoted above [11,12,25–27] and may be attributed to the presence of Te$\cdots$O secondary bonds [28].

As discussed above (Section 3.3.1) in the case of $C_5H_{10}Te(OCOC_6H_5)_2$ (2), the Te$\cdots$O secondary bond in $C_8H_8Te(OCOC_6H_5)_2$ (7) brings up the coordination of the tellurium 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 atom (the secondary bond itself is in the *trans* position to the other –CH$_2$ group attached to tellurium). The angles TeA–O(2A)–C(1) and TeA–O(2A)–C(8) are 160.6 and 74.6° (Table 5). Similar coordination is shown in the other organotellurium complexes [12,27,29] in which the unshared electron pair is in the *trans* position to the organic group. The Te$\cdots$O (3.376 Å) corresponds to a secondary bond [28] and is comparable to the Te$\cdots$O secondary bond in 2. These weak Te$\cdots$O secondary bonds join together four $C_8H_8Te(OCOC_6H_5)_2$ (7) molecules (Fig. 4) resulting in tetrameric (stair like) supramolecular associations whereas in the precursor ($C_8H_8TeI_2$) three dimensional polymers [3] are present.

3.3.3. Supramolecular associations

On comparing the supramolecular associations present in $C_8H_8Te(OCOC_6H_5)_2$ (7), $C_5H_{10}Te(OCOC_6H_5)_2$ (2) [present work] and earlier reported $C_4H_8Te(OCOC_6H_5)_2$ [12] vis-a-vis in their precursors $C_8H_8TeI_2$, $C_5H_{10}TeI_2$, $C_4H_8TeI_2$, [3], it is observed that in 7 tetrameric (stair like) supramolecular associations, directed through Te$\cdots$O secondary bonds, are present (Fig. 4). Te$\cdots$O bond length is 3.376 Å. The H$_2$C(8)–Te, H$_2$C(1)–Te bond lengths are 2.106(2) and 2.111(2) Å

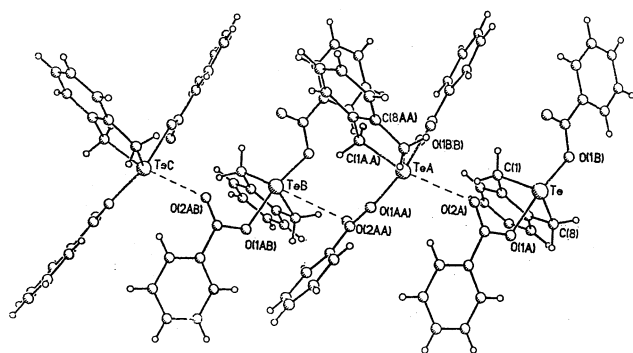
Fig. 4. Molecules of 7 connected through Te$\cdots$O secondary bonds (broken).
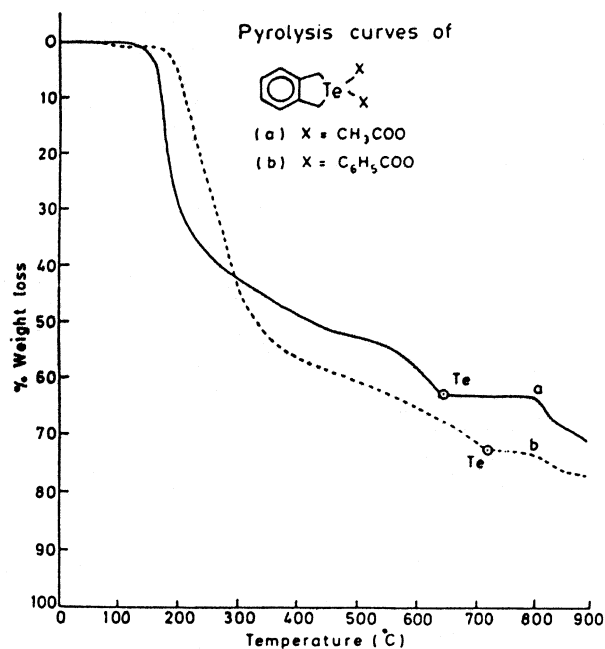

Fig. 5. (a) Pyrolysis curve of 6. (b) Pyrolysis curve of 7.

respectively. The C–C bond lengths in C_6H_4, cycle are of the order of 1.385 Å. In the precursor $C_8H_8TeI_2$, three dimensional polymers are present linked through Te$\cdots$I secondary bonds. Te$\cdots$I bond length is 2.902(7) Å. The CH$_2$–Te bond lengths are of the order of 1.38 Å [3]. In 2, Te$\cdots$O secondary bonds directed polymeric (*zig-zag chains*) supramolecular associations are present. The Te$\cdots$O bond length is 3.307 Å. The H$_2$C(1)–Te, H$_2$C(5)–Te bond lengths are 2.127(2) and 2.132(2) Å, respectively and C–C bond lengths of $C_5H_{10}Te$ cycle are of the order of 1.525(3) Å. In the precursor $C_5H_{10}Te_2$, trimeric aggregates are present; the units are linked through Te$\cdots$I secondary bonds (4.065, 3.870 Å) and I$\cdots$I (3.722 Å) secondary bonds. The CH$_2$–Te bond lengths and C–C bond lengths in $C_5H_{10}Te$ cycle are of the order of 2.131(1) and 1.43(4) Å, respectively. In $C_4H_8Te(OCOC_6H_5)_2$, *zig-zag* polymeric chain based supramolecular associations are directed through Te$\cdots$O secondary bonds. The Te$\cdots$O secondary bond distance is 3.083 Å. The CH$_2$–Te bond lengths 2.119(4) Å and C–C bond lengths are of the order of 1.519 Å [12]. In the precursor $C_4H_8TeI_2$, the *zig-zag* two dimensional ribbons, interlinked through Te$\cdots$I secondary bonds, arc present. The Te$\cdots$I bond length is 3.808 Å. The CH$_2$–Te bond lengths are 2.157(7) Å and C–C bond lengths are of the order of 1.51(2) Å [3].

These results demonstrate that the change of iodide with benzoate group leads to:

a) Tellurium is hexa- and hepta-coordinated in the precursors whereas $C_8H_8Te(OCOC_6H_5)_2$ (7) contains hexacoordinated tellurium, $C_5H_{10}Te$-

$(OCOC_6H_5)_2$ (**2**) and $C_4H_8Te(OCOC_6H_5)_2$ [12] contain heptacoordinated tellurium.

b) There is no change in the conformation of tellurium heterocycles (C_8H_8Te, $C_5H_{10}Te$ and C_4H_8Te).

c) The polymeric supramolecular association of $C_8H_8TeI_2$ is changed into tetrameric (*stair like*) supramolecular association in $C_8H_8Te(OCOC_6H_5)_2$ (**7**).

d) The trimeric supramolecular aggregates in $C_5H_{10}TeI_2$ are changed into polymeric (*zig-zag chain*) supramolecular associations in $C_5H_{10}Te(O-COC_6H_5)_2$ (**2**).

e) The *zig-zag ribbon* based supramolecular association in $C_4H_8TeI_2$ is changed into polymeric *zig-zag chain* based supramolecular association in $C_4H_8Te(OCOC_6H_5)_2$ [12].

4. Supplementary material

Crystallographic data for the structural analysis (excluding structure factors) has been deposited with the Cambridge Crystallographic Data Center, CCDC nos. 138546 and 170225 for compounds **2** and **7**. 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; email: deposit@ ccdc.cam.ac.uk or www:http://www.ccdc.cam.ac.uk).

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