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Synthesis and crystal structures of the monomeric organotellurium(IV) trihalides: *trans*-2-ethoxy-cyclohexyl-tellurium(IV) trichloride, trichloro-(2-chlorobicyclo[2.2.1]hept-7-yl)-λ^4-tellurane, and mesityltellurium(IV) tribromide

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Abstract

The crystal and molecular structures of *trans*-2-ethoxy-cyclohexyltellurium(IV) trichloride (**1**), trichloro(2-chlorobicyclo[2.2.1]hept-7-yl)-λ^4-tellurane (**2**) and mesityltellurium(IV) tribromide (**3**) have been determined. The structures of **1** and **2** show intramolecular bonding between additional donors resident on the organogroups and the tellurium(IV) centre leading to pseudo-octahedral geometries. The structure of **3** shows no evidence of intra- or inter-molecular bonding, and has clearly a five-coordinate geometry about the tellurium centre, indicating the presence of a stereochemically active lone pair in a trigonal bipyramidal arrangement of electron pairs. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Tellurium; X-ray structure; Haloalkyls; Haloaryls

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1. Introduction

VSEPR theory suggests that a tellurium(IV) centre bonded to four ligands should have four of the six valence electrons used in bonding and the two remaining electrons as a lone pair. Consequently, the tellurium(IV) centre should be surrounded by five electron-pairs and theoretically have a trigonal bipyramidal geometry in which the lone pair occupies an equatorial position. Crystallographic studies of diorganotellurium(IV) dihalides, R_2TeX_2, have shown that in the solid state they have trigonal bipyramidal geometries as predicted by VSEPR theory [1]. However, the organotellurium(IV) trihalides, $RTeX_3$, generally have octahedral geometries in the solid state owing to the intermolecular association between mononuclear units thereby indicating the presence of other factors, such as the Lewis acidity of the tellurium(IV) centre, which affect the coordination geometry.

For most organotellurium(IV) trihalide crystal structures reported [1–23], the lone pair is stereochemically active, that is, the electron pair participates in the hybridisation of the tellurium(IV) centre and thereby occupies an effective stereochemical position. However, in every case reported, there are additional secondary bonding interactions that affect the overall geometry about the tellurium(IV) centre. In this context, secondary bonding can be considered as an interaction that is longer than the sum of the respective covalent radii, but shorter than the sum of the van der Waals radii [24].

The structures of organotellurium(IV) trihalides can be divided into two general groups. The first group includes those compounds which contain intermolecular secondary bonding interactions and are oligomeric, or in some cases, polymeric. The second group of organotellurium(IV) trihalides is monomeric, due to the secondary intramolecular interactions.

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In continuation of our earlier investigations on the stereochemical activity of lone pairs in tellurium(IV) complexes [25–31], we now report what appears to be the first example of a monomeric organotellurium(IV) trihalide with no intramolecular bonding, mesityltellurium(IV) tribromide (**3**). The geometry about the tellurium(IV) centre is trigonal bipyramidal with a stereochemically active lone pair. The structures of *trans*-2-ethoxy-cyclohexyltellurium(IV) trichloride (**1**) and trichloro(2-chlorobicyclo[2.2.1]hept-7-yl)-λ^4-tellurane (**2**) are monomeric, but in these examples, intramolecular bonding between the tellurium centre and the donor substituents in the organic groups give rise to pseudo octahedral geometries.

2. Experimental

2.1. Preparation of *trans*-2-ethoxy-cyclohexyltellurium-(IV) trichloride $(2\text{-}CH_3CH_2O)C_6H_{10}TeCl_3$ (*1*)

The literature method describing preparation of *trans*-2-ethoxycyclohexyltellurium(IV) trichloride [32] gave a mixture of the desired product and the *trans*-2-chlorocyclohexyltellurium(IV) trichloride. So a slight modification of the method was used to obtain a pure product in a higher yield. Tellurium tetrachloride (20 g) was refluxed in dried ethanol (100 ml) with cyclohexene (15 ml) for 2 h and then filtered while still warm and allowed to cool to room temperature (r.t.). The product was collected and recrystallised from 40 to 60 pet. spirits to yield almost colourless needles, m.p. 95–100°C (lit. 97–98°C). ^{1}H-NMR δ 4.4 (ddd, 1H, CHTe), 4.25 (ddd, 1H, CHO), 3.86 (dq, 1H, CH_2O), 2.53, 2.20, 2.10, 1.40 (broad m, 8H, CH_2), 1.24 (dd, 3H, CH_3); δ^{13}C 79.7 $^1J(^{13}\text{C-}^{125}\text{Te})$ 158 Hz, 79.2, 65.2, 32.9, 27.4, 25.6, 23.1, 15.1 ppm; δ^{125}Te −146 ppm (toluene).

2.2. Preparation trichloro(2-chlorobicyclo[2.2.1]-hept-7-yl)-λ^4-tellurane (*2*)

Compound **2** was prepared by dissolving equimolar quantities of norbornylene (1.8 g) and tellurium(IV) tetrachloride (5 g) in carbon tetrachloride (50 ml). The mixture was refluxed for 3 h and then the warm reaction mixture was filtered and allowed to cool to r.t. The product was collected and recrystallised from hot carbon tetrachloride to give colourless needles (3.5 g, 52%), m.p. 135–140°C, dec. ^{1}H-NMR 4.60 (s, 1H, H7), 4.46 (d, 1H, H2), 3.65 (d, 1H, H1), 3.40 (dd, 1H, H4), 2.77 (d, 1H, H3 *exo*), 2.14 (m, 1H, H3 *endo*), 2.06 (m, 1H, H6 *exo*), 1.8 (m, 1H, H5 *exo*), 1.6 (m, 2H, H5, H6 *endo*); ^{13}C 78.56, 67.3, 50.6, 40.6, 40.5, 28.5, 28.4 ppm. ^{125}Te −269 ppm (CH_2Cl_2). Microanalysis found: C, 22.91; H, 2.64; Br, 39.49%. $C_7H_{10}Cl_4Te$ requires C, 23.13; H, 2.77; Br, 39.01%.

2.3. Preparation of mesityltellurium(IV) tribromide, $2,4,6\text{-}(CH_3)_3C_6H_2TeBr_3$ (*3*)

A solution of bromine (18 g) in dichloromethane was added to dimesityl ditelluride (18.5 g) [33] in dichloromethane cooled in an ice bath. The addition of bromine was continued until the red colour of the ditelluride disappeared indicating that the reaction was complete. Solvent and excess bromine were removed by rotary evaporation. The resultant orange solid was recrystallised from 60 to 80 pet. spirits to give orange crystals (24 g, 66%), m.p. 128–130°C. ^{1}H-NMR 6.88 (s, 2H, Ar) 2.37 (s, 6H, 2-CH_3), 2.23 (s, 3H, 4-CH_3); ^{125}Te −469 ppm, (toluene). Microanalysis found: C, 22.51; H, 2.09; Br, 49.26%. $C_9H_{11}Br_3Te$ requires C, 22.22; H, 2.28; Br, 49.27%.

2.4. Crystallography

Intensity data were collected at r.t. on a Rigaku AFC6R diffractometer (Enraf–Nonius CAD4 for **3**) fitted with graphite monochromatised Mo–K$_\alpha$ radiation and the $\omega{:}2\theta$ scan technique. Data were corrected routinely for Lorentz and polarisation effects and for absorption employing empirical (**1** and **2** [34]) and analytical (**3** [35]) procedures. The structures were solved by direct-methods [36] and each refined by a full-matrix least-squares procedure based on F [37,35]. Non-hydrogen atoms were refined with anisotropic displacement parameters and hydrogen atoms were included in the models at their calculated positions. After the introduction of a weighting scheme of the form $w = 1/[\sigma^2(F) + g|F|^2]$, each refinement was continued until convergence. Crystallographic data and final refinement details are collected in Table 1, and the numbering schemes employed are shown in Figs. 1–3 which were drawn with ORTEP [38] at the 35% probability level.

3. Results and discussion

3.1. Description of the structure of $EtOC_6H_{10}TeCl_3$ (*1*)

The molecular structure of **1** is shown in Fig. 1 and relevant bond lengths and angles are listed in Table 2. Two enantiomeric stereoisomers (the C(1) and C(2) atoms are chiral) of **1** comprise of the crystallographic asymmetric unit. The *S*,*S* isomer (molecule *a*) is shown in Fig. 1, the *R*,*R* isomer has a similar numbering scheme. A comparison of the geometric parameters defining the molecular geometries reveals that there are several crystallographically significant differences between the independent molecules. Differences between the molecules relate, in the main, to the Te–Cl bond distances that can be rationalised in terms of inter-

Table 1
Summary of crystal data for $EtOC_6H_{10}TeCl_3$ (**1**), $2\text{-}ClC_7H_{10}TeCl_3$ (**2**) and $2,4,6\text{-}Me_3C_6H_2TeBr_3$ (**3**)

Compound	**1**	**2**	**3**
Formula	$C_8H_{15}Cl_3OTe$	$C_7H_{10}Cl_4Te$	$C_9H_{11}Br_3Te$
Formula weight	361.2	363.6	486.5
Crystal size (mm)	0.10 × 0.16 × 0.32	0.03 × 0.15 × 0.30	0.03 × 0.03 × 0.08
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1/c$	$P\bar{1}$	$P2_1/c$
a (Å)	12.186(3)	7.981(3)	8.151(1)
b (Å)	8.538(2)	11.315(3)	21.475(2)
c (Å)	24.448(2)	6.887(2)	7.451(1)
α (°)	90	94.27(3)	90
β (°)	91.26(1)	114.40(3)	97.25(1)
χ (°)	90	102.96(3)	90
V (A^3)	2543.1(7)	542.1(4)	1293.8(2)
Z	8	2	4
$D_{calc.}$ (g cm^{-3})	1.886	2.227	2.497
$F(000)$	1392	344	888
μ (cm^{-1})	29.34	36.73	115.51
Trans factors	0.760–1	0.637–1	0.341–0.635
No. of data collected	6775	2764	3315
No. of unique data	6480	2579	2937
Data with $I \geq 3.0\sigma(I)$	3217	2026	1148 [a]
R	0.040	0.031	0.051
g	0.00006	0.00002	0.0057
R_w	0.039	0.032	0.055
Residual electron density (e A^{-3})	0.80	0.73	1.42

[a] Data with $I \geq 2.5\sigma(I)$.

molecular Te···Cl interactions, as discussed below. In addition, there are some differences in the bond angles subtended at the tellurium atoms with the greatest of ca. 8° being found for the Cl(2)–Te–O(1) angle.

The oxygen atom in **1** forms a significant intramolecular Te···O interaction of 2.657(4) Å (2.643(5) Å for molecule *b*). Although these distances are greater than the sum of their covalent radii (2.07 Å [24]), they are considerably shorter than the sum of their van der Waals radii (3.60 Å [24]). Thus, the tellurium atom is best described as having a geometry that is intermediate between trigonal bipyramidal and octahedral, where the lone pair occupies the vacant site in each case. In this sense, the geometry about the tellurium atom in **1** is typical of the monomeric structures found for organotellurium(IV) trihalides. The axial chlorine atoms and the tellurium are almost linear, the angle Cl(1)*a*–Te(1)–Cl(2)*a* for the *S,S* isomer (molecule *a*) is 177.86(7)° (176.34(9)° for molecule *b*) and form significantly longer bonds than does the equatorially bound chloride. Not surprisingly, in terms of the ligand donor set the greatest distortion from the ideal octahedral geometry may be traced to the restricted bite angles of the C–, O–chelate (56.0(2) and 56.1(2)°, respectively). Two very similar compounds reported in the literature, 8-ethoxy-4-cyclooctenyltellurium(IV) trichloride [14] and *cis*-2-ethoxycycloheptyltellurium(IV) tribromide [20] have Te–O bond lengths of 2.419(2) and 2.49(3) Å, respectively.

As mentioned above, there are some significant differences between the Te–Cl bond distances. Thus, Te–Cl(1), Te–Cl(2) and Te–Cl(3) are 0.04 Å longer, 0.06 Å shorter and 0.04 Å longer for molecule *a* compared with molecule *b*. These differences may be accounted for in terms of intermolecular Te···Cl interactions. Thus, Cl(1)*a* forms a contact of 3.605(2) Å

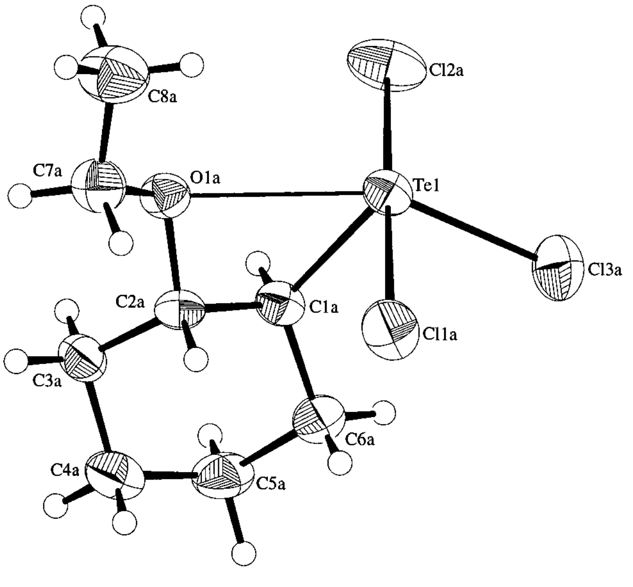

Fig. 1. Molecular structure for molecule *a* of $EtOC_6H_{10}TeCl_3$ (**1**); the numbering scheme for molecule *b* is similar.

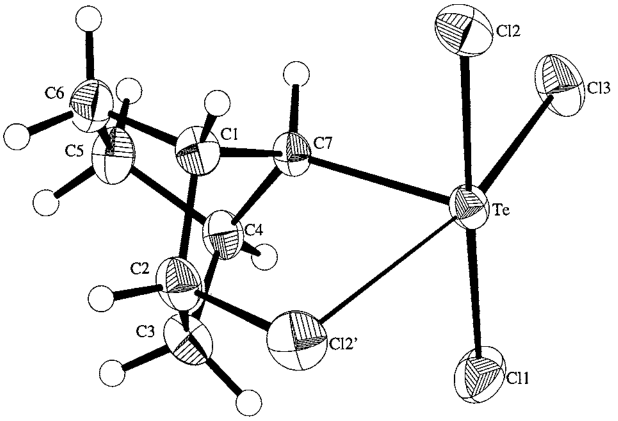

Fig. 2. Molecular structure of 2-$ClC_7H_{10}TeCl_3$ (**2**).

Table 2
Selected geometric parameters (Å, °) for $EtOC_6H_{10}TeCl_3$ (**1**)

	Molecule *a*	Molecule *b*
Bond lengths		
Te–Cl(1)	2.481(2)	2.445(3)
Te–Cl(2)	2.433(2)	2.490(3)
Te–Cl(3)	2.300(2)	2.265(3)
Te–C(1)	2.140(6)	2.133(7)
Te–O(1)	2.657(4)	2.643(5)
Bond angles		
Cl(1)–Te–Cl(2)	177.86(7)	176.34(9)
Cl(1)–Te–Cl(3)	88.38(8)	90.1(1)
Cl(1)–Te–C(1)	90.1(2)	87.9(2)
Cl(1)–Te–O(1)	86.0(1)	92.3(1)
Cl(2)–Te–Cl(3)	90.14(8)	89.2(1)
Cl(2)–Te–C(1)	88.6(2)	88.7(2)
Cl(2)–Te–O(1)	94.6(1)	86.8(1)
Cl(3)–Te–C(1)	97.5(2)	98.9(2)
Cl(3)–Te–O(1)	152.8(1)	154.7(1)
C(1)–Te–O(1)	56.0(2)	56.1(2)
Te–C(1)–C(2)	102.3(4)	102.4(5)
Te–C(1)–C(6)	121.1(5)	122.6(5)
Te–O(1)–C(2)	82.3(3)	82.7(3)
Te–O(1)–C(7)	129.3(4)	128.7(4)
C(2)–O(1)C(7)	115.0(5)	117.5(6)

with a centrosymmetrically related molecule leading to the formation of loosely associated dimers. The Te···Cl(1)a^{i} distance is less than the sum of their van der Waals radii of 3.90 Å [24]; symmetry operation (i): $1-x$, $1-y$, $-z$. Similarly, the Cl(2)*b* atom is involved in a contact with a symmetry related Te(2) atom such that Te(2)···Cl(2)b^{ii} is 3.613(3) Å; symmetry operation (ii): $-x$, $0.5+y$, $0.5-z$. This type of interaction leads to the formation of chains of molecule *b* throughout the crystal lattice, aligned along the crystallographic *b*-direction. Thus, the elongation of the Te(1)–Cl(1)*a* and Te(2)–Cl(2)*b* distances with respect to the other molecules may be related directly to the fact that the Cl(1)*a* and Cl(2)*b* atoms participate in intermolecular Te···Cl interactions. Less obvious, however, is the reason for the disparity in the Te–Cl(3) distances between the two independent molecules. An examination of the conformation found for the loosely associated dimer formed by molecules of *a*, shows that each lone pair of electrons is directed towards the position occupied by a Cl(3) atom and hence, provides a plausible explanation for the observed difference.

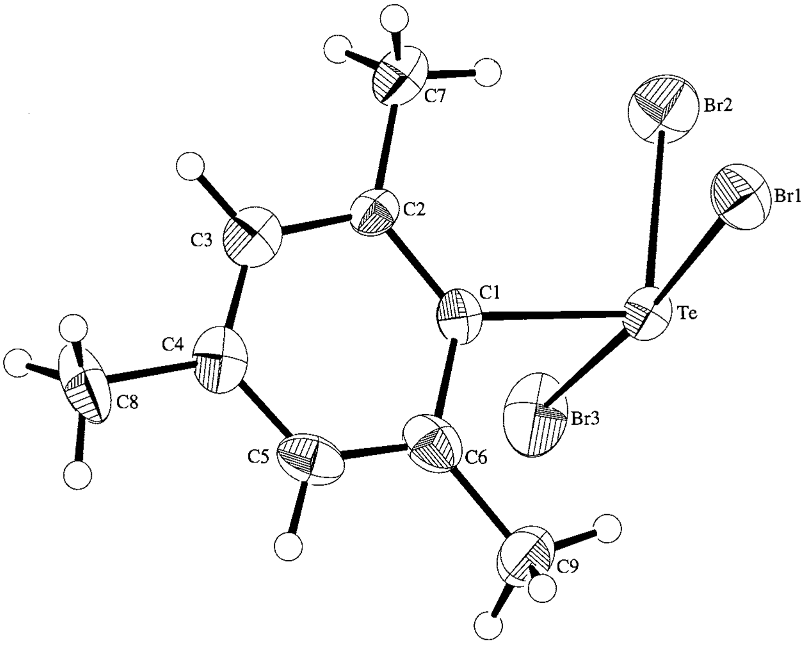

Fig. 3. Molecular structure of 2,4,6-$(CH_3)_3C_6H_{12}TeBr_3$ (**3**).

*3.2. Description of the structure of 2-ClC$_7$H$_{10}$TeCl$_3$ (**2**)*

The molecular structure of **2** is shown in Fig. 2 and selected bond distances and angles are given in Table 3. The molecular geometry is similar to that found for **1** except that the intramolecular donor in this case is a chloride, attached to the norbornyl group. The Te–Cl(2′) distance is 3.013(2) Å and the C(7)–Te–Cl(2′) chelate angle is 73.6(1)°. Centrosymmetrically related molecules of **2** also associate to form loosely bound dimers as found for **1**. The Te–Cl(2)^i distance of 3.711(2) (symmetry operation (i): $-x$, $-y$, $-z$) is marginally longer than that found in **1**. However, in **2** there is no significant disparity in the Te–Cl$_{axial}$ bond distances as found in **1**. A close inspection of intramolecular Cl···H contacts in **2** reveals that the Cl(1) atom is involved in two contacts < 3.0 Å and that the Cl(2) atom is only involved in one such interaction. The importance of considering Cl···H contacts in rationalising otherwise unexplained differences in geometric parameters in diorganotin(IV) systems has been highlighted in recent combined crystallographic/theoretical studies [39,40].

*3.3. Description of the structure of 2,4,6-(CH$_3$)$_3$C$_6$H$_{12}$TeBr$_3$ (**3**)*

The molecular structure of **3**, shown in Fig. 3, matches that expected from VSEPR as there are no significant intra- or intermolecular interactions present; selected interatomic parameters are given in Table 4. The lack of significant additional interactions is consistent with the reduced Lewis acidity of the tellurium atom in CTeBr$_3$ compared with that in the CTeCl$_3$ donor set. The geometry is clearly based on a trigonal bipyramid with the lone pair of electrons occupying a position in the equatorial plane. The most significant deviation from the ideal geometry is found in the magnitude of the Br(2)–Te–C(1) angle of 107.8(6)°, clearly consistent with the presence of the lone pair in the equatorial plane. The difference between the Te–Br distances formed by the axially- and equatorially-bound bromide atoms matches the expected trend and the small disparity in the Te–Br$_{axial}$ distances may, again, be traced to the presence of loosely associated centrosymmetric dimers in the solid state. Thus, the Te···Br(3)^i separation is 3.815(8) Å, a distance within 4.0 Å, being the sum of their van der waals radii [24]; symmetry operation (i): $1-x$, $1-y$, $1-z$.

Table 3
Selected geometric parameters (Å, °) for 2-ClC$_7$H$_{10}$TeCl$_3$ (**2**)

Bond lengths			
Te–Cl(1)	2.462(2)	Te–Cl(2)	2.467(2)
Te–Cl(3)	2.299(2)	Te–C(7)	2.144(5)
Te–Cl(2′)	3.013(2)		
Bond angles			
Cl(1)–Te–Cl(2)	175.98(6)	Cl(1)–Te–Cl(3)	88.42(7)
Cl(1)–Te–C(7)	93.3(1)	Cl(1)–Te–Cl(2’)	92.80(6)
Cl(2)–Te–Cl(3)	87.57(7)	Cl(2)–Te–C(7)	86.6(2)
Cl(2)–Te–Cl(2′)	91.01(6)	Cl(3)–Te–C(7)	92.8(1)
Cl(3)–Te–Cl(2′)	166.41(6)	C(7)–Te–Cl(2’)	73.6(1)

Table 4
Selected geometric parameters (Å, °) for 2,4,6-Me$_3$C$_6$H$_2$TeBr$_3$ (**3**)

Bond lengths			
Te–Br(1)	2.641(2)	Te–Br(2)	2.471(4)
Te–Br(3)	2.683(3)	Te–C(1)	2.13(2)
Bond angles			
Br(1)–Te–Br(2)	91.0(1)	Br(1)–Te–Br(3)	174.8(4)
Br(1)–Te–C(1)	89.2(4)	Br(2)–Te–Br(3)	85.9(1)
Br(2)–Te–C(1)	107.8(6)	Br(3)–Te–C(1)	87.7(4)

4. Conclusions

It appears from the results of X-ray crystal structure determinations that the tellurium(IV) centre in these compounds prefers a coordination number of at least six (i.e. five donors and one lone pair), which it attains either by intra- or intermolecular bonding depending on the nature of the ligands present. This requirement for additional electron density is a result of the residual Lewis acidity of the tellurium in organotellurium(IV) trihalides, created by the presence of three strongly electron withdrawing groups. Significant intermolecular association may be curtailed by the presence of bulky organic substituents bound to tellurium, however, intramolecular interactions may be formed when additional potential donor atoms are present on the organic groups. The combination of a bulky organic substituent with reduced Lewis acidity at the tellurium centre, as in **3**, allows the formation of monomeric species with structures consistent with the VSEPR model.

5. Supplementary material

The crystallographic details have been deposited as Crystallographic Information Files at the Cambridge Crystallographic Data Centre with deposition numbers: 132386–132388. Tables of observed and calculated structure factors are available on request (e-mail: edward.tiekink@adelaide.au).

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