

## PREPARATION AND CRYSTAL STRUCTURES OF TRIETHYLTELLURONIUM CHLORIDE AND IODIDE

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### Summary

Triethyltelluronium chloride and triethyltelluronium iodide have been synthesized and their structures determined by X-ray crystallography. Crystals of  $\text{Et}_3\text{TeCl}$  are cubic, space group  $I\bar{4}3m$ ,  $a$  12.383(4) Å,  $V$  1899(1) Å<sup>3</sup> and  $Z = 8$  and those of  $\text{Et}_3\text{TeI}$  are monoclinic, space group  $P2_1/n$ ,  $a$  7.404(2),  $b$  12.780(3),  $c$  11.163(3) Å,  $\beta$  90.57(2)°,  $V$  1056.2(5) Å<sup>3</sup> and  $Z = 4$ . In  $\text{Et}_3\text{TeCl}$ , the tellurium and chlorine atoms form isolated cubane-type tetramers with  $T_d$  symmetry where the monomeric units are held together by secondary interactions. The structure of  $\text{Et}_3\text{TeI}$  consists of pairs of triethyltelluronium cations which are combined through pairs of bridging iodide ions. Much longer Te --- I interactions between the dimers result in the occupation of the sixth position of a distorted octahedron around the tellurium atom.

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### Introduction

Crystal structures of a few triorganotelluronium salts have been determined [1–4]. All of the compounds, with the exception of  $\text{Me}_3\text{Te}^+ \text{BF}_4^-$  [4], show cation–anion interactions. We have recently published the structure of a telluronium halide, namely  $\text{Et}_3\text{TeBr}$  [5], in which each tellurium atom is involved in three weak secondary bonds with bromine atoms to form a cubane-type tetramer. We decided to study the X-ray structure of the chloride and iodide to compare the structural features of the three halides.

### Experimental

$\text{Et}_2\text{Te}$  and  $\text{EtI}$  were purchased from Strem Chemicals Inc. and the Fisher Scientific Co. respectively and used as supplied.  $\text{Ag}_2\text{O}$  was prepared from  $\text{AgNO}_3$  and  $\text{NaOH}$ . IR spectra were recorded in Nujol mulls on a Perkin–Elmer 180 spectrophotometer in polyethylene disks. <sup>1</sup>H NMR spectra were recorded at 60 MHz

using a Varian EM-360 instrument with TMS as internal standard. Carbon and hydrogen microanalyses were performed by Guelph Chemical Laboratories Ltd. Tellurium and halogen were determined using standard gravimetric techniques. The density was measured by the flotation method in  $\text{CCl}_4/\text{CH}_2\text{I}_2$ .

### Synthesis of compounds

#### *Triethyltelluronium iodide*

Triethyltelluronium iodide was prepared by treating  $\text{Et}_3\text{Te}$  with excess of  $\text{EtI}$  and keeping the solution overnight in a refrigerator. The excess of  $\text{EtI}$  was then decanted and colorless needles of  $\text{Et}_3\text{TeI}$  were then washed with ether and dried in vacuo. Recrystallization from  $\text{CH}_2\text{Cl}_2$  gave  $\text{Et}_3\text{TeI}$ , m.p.  $93^\circ\text{C}$ , lit.  $92^\circ\text{C}$  [6]. Anal. Found: C, 21.00; H, 4.51; Te, 37.90; I, 36.50;  $\text{C}_6\text{H}_{15}\text{TeI}$  calcd.: C, 21.08; H, 4.39; Te, 37.35; I, 37.18%. IR ( $\text{cm}^{-1}$ ): 504 ( $\nu(\text{Te}-\text{C})$ ), 300m, 280m, 244w, 225w, 180m, 155w.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  1.58 (t, 3H), 3.00 (q, 2H).  $J(\text{CH}_2-\text{CH}_3)$  8 Hz.

#### *Triethyltelluronium chloride*

Triethyltelluronium iodide (2.5 g) was ground up with  $\text{Ag}_2\text{O}$  (2.0 g) and a few drops of water in a mortar for 10–15 min. The slurry was stirred with water (15 ml) for half an hour and filtered. Treatment of the filtrate with 4*N* hydrochloric acid (5 ml) gave  $\text{Et}_3\text{TeCl}$  in aqueous medium, which was extracted twice with  $\text{CH}_2\text{Cl}_2$  (10 ml). Slow evaporation of the organic layer after drying over anhydrous  $\text{CaCl}_2$  gave crystals of  $\text{Et}_3\text{TeCl}$  (1.4 g, 75% yield), m.p.  $184^\circ\text{C}$ . Anal. Found: C, 28.91; H, 6.40; Te, 50.15; Cl, 14.45.  $\text{C}_6\text{H}_{15}\text{TeCl}$  calcd.: C, 28.79; H, 6.00; Te, 51.02; Cl, 14.19%. IR ( $\text{cm}^{-1}$ ): 502 ( $\nu(\text{Te}-\text{C})$ ), 300w, 290w, 175w.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  1.62 (t, 3H), 3.15 (q, 2H).  $J(\text{CH}_2-\text{CH}_3)$  8 Hz.

### *Crystallographic analysis*

X-ray diffraction results for both the compounds were collected on a Syntex P2<sub>1</sub> diffractometer by procedures already described [7]. A summary of the crystal data is given in Table 1. The intensities of three standard reflections monitored at intervals of 50 reflections fluctuated by less than 2% during data collection. Cell parameters of both the compounds were refined from 15 high angle ( $15 < \theta < 30^\circ$ ) strong reflections. Each set of data was corrected for Lorentz and polarization effects and analytical absorption corrections were applied.

For  $\text{Et}_3\text{TeCl}$ , the space group  $I\bar{4}3m$  was established from axial photographs showing  $m\bar{3}m$  Laue symmetry, the systematic absences ( $hkl$ ,  $h + k + l = 2n + 1$ ) and Patterson vectors, which required the tellurium and chlorine atoms to be on  $3m$  site symmetry. The compound is thus isomorphous with  $\text{Et}_3\text{TeBr}$  [5]. This suggested that the atomic coordinates of tellurium and chlorine should be approximately the same as those of the analogous atoms in  $\text{Et}_3\text{TeBr}$ . The two carbon atoms were found to be disordered across the mirror plane with a small separation ( $\text{C}(1) \cdots \text{C}(1)$  0.86 and  $\text{C}(2) \cdots \text{C}(2)$  1.05 Å). Therefore, the carbon atoms were refined isotropically while tellurium and chlorine atoms were refined anisotropically. The convergence was achieved at  $R = 0.0447$  and  $R_w = 0.0499$ .

For the compound  $\text{Et}_3\text{TeI}$ , the space group  $P2_1/n$  was identified from systematic absences  $0k0$ ,  $k = 2n + 1$ ,  $h0l$ ,  $h + l = 2n + 1$ . The positions of tellurium and iodine

TABLE 1

SUMMARY OF CRYSTAL DATA<sup>a</sup>, INTENSITY COLLECTION, AND STRUCTURAL REFINEMENT FOR Et<sub>3</sub>TeCl (A) AND Et<sub>3</sub>TeI (B)

	A	B
Cell constants	$a$ 12.383(4) Å <sup>b</sup>	$a$ 7.404(2), $b$ 12.780(3) $c$ 11.163(3) Å, $\beta$ 90.57(2)°
Cell volume (Å <sup>3</sup> )	1899(1)	1056.2(5)
Crystal system	cubic	monoclinic
Space group	$I\bar{4}3m$	$P2_1/n$
Mol. wt.	250.1	341.6
Z	8	4
Crystal dimensions (mm)	0.23 × 0.25 × 0.27	0.15 × 0.19 × 0.31
$\rho_c$ , $\rho_0$ (g cm <sup>-3</sup> )	1.75, 1.73	2.15, 2.18
Abs coeff, $\mu$ (cm <sup>-1</sup> )	31.04	52.95
Min abs. corr	1.725	2.328
Max abs. corr	1.982	3.085
Radiation		Mo-K $\alpha$ , $\lambda$ 0.71069 Å
Monochromator		highly oriented graphite
Temp. (°C)		21
2 $\theta$ angle (°)	4–50	4–45
Scan type		coupled $\theta$ (crystal)/2 $\theta$ (counter)
Scan width		$K_{\alpha_1} - 1^\circ$ to $K_{\alpha_2} + 1^\circ$
Scan speed (° min <sup>-1</sup> )		variable, 2.02–4.88
Bkgd time/scan time		0.5
Total reflns. measd.	726 (+ $h$ , + $k$ , + $l$ )	1785 (+ $h$ , + $k$ , $\pm$ $l$ )
Uniq. data used	157 [ $I > 3\sigma(I)$ ]	976 [ $I > 3\sigma(I)$ ]
No. of parameters (NP)	15	68
$R = (\sum   F_0  -  F_c   / \sum  F_0 )$	0.0447	0.0384
$R_w = [\sum w( F_0  -  F_c )^2 / \sum w F_0 ^2]$	0.0499	0.0503
$\Delta\rho_{\max}$ (e Å <sup>-3</sup> )	0.7	0.7
Shift: error (max)	0.02	0.2

<sup>a</sup> Standard deviations in parentheses. <sup>b</sup> Et<sub>3</sub>TeBr,  $a$  12.595(4) Å.

atoms were obtained from direct methods using Shelx. The positions of carbon atoms were determined from a difference map. Anisotropic refinement of all atoms gave a chemically unreasonable Te–C(5)–C(6) bond angle as well as Te–C(5) and C(5)–C(6) distances so that it appeared that there was disorder. However, when the Te–C(5) and C(5)–C(6) distances were constrained at 2.14 and 1.44 Å respectively, the refinement resulted in reasonable bond lengths and angles with two C(6) positions having 75 and 25% occupancy. In the final cycles of refinement, these atoms were refined isotropically while all others were refined anisotropically. The refinement converged to  $R = 0.0384$  and  $R_w = 0.0503$ .

No attempt was made to include hydrogen atoms in either of the two structures. In both cases the function minimized in the least-squares refinement was  $(|F_0| - |F_c|)^2$ . Unit weights were used in the initial stages, while in the final cycles, a weighting scheme of the form  $\omega = 1/[\sigma^2(F) + 0.0001F^2]$  was employed. Computer programs and the sources of scattering factors were those reported previously [8]. Positional and thermal parameters are given in Table 2 and interatomic distances and angles in Table 3. Structure factor tables may be obtained from the authors.

TABLE 2  
FINAL POSITIONAL AND THERMAL PARAMETERS (Å<sup>2</sup>) FOR NON-HYDROGEN ATOMS WITH THEIR STANDARD DEVIATIONS IN PARENTHESES<sup>a</sup>

	x	y	z	$U_{11}$	$U_{22}$	$U_{33}$	$U_{12}$	$U_{13}$	$U_{23}$
<i>A. Et<sub>2</sub>TeCl</i>									
Te	0.1296(1)	0.1296(1)	0.1296(1)	0.0754(8)	0.0754(8)	0.0754(8)	-0.0026(6)	-0.0026(6)	-0.0026(6)
Cl	0.3523(4)	0.3523(4)	0.3523(4)	0.092(3)	0.092(3)	0.092(3)	0.011(2)	0.011(2)	-0.011(2)
C(1) <sup>a</sup>	0.113(3)	0.299(3)	0.162(2)	0.111(12)					
C(2) <sup>a</sup>	0.136(4)	0.370(4)	0.076(5)	0.174(23)					
<i>B. Et<sub>2</sub>TeI</i>									
I	0.1302(1)	-0.1345(1)	0.1975(1)	0.0889(8)	0.0878(7)	0.0900(8)	0.0038(5)	0.0113(6)	0.0032(5)
Te	0.0378(1)	0.1437(1)	0.1433(1)	0.0684(7)	0.0769(7)	0.0764(7)	-0.0030(4)	0.0113(5)	0.0132(4)
C(1)	0.270(2)	0.042(1)	0.129(1)	0.077(10)	0.108(10)	0.094(10)	-0.011(8)	-0.0031(8)	0.000(8)
C(2)	-0.439(3)	0.083(2)	0.158(2)	0.101(14)	0.127(15)	0.257(28)	-0.035(12)	0.039(16)	-0.034(15)
C(3)	-0.168(3)	0.289(1)	0.115(2)	0.147(15)	0.076(9)	0.172(16)	0.025(10)	0.074(12)	0.025(10)
C(4)	-0.061(3)	0.369(1)	0.056(2)	0.153(17)	0.092(11)	0.112(13)	-0.007(10)	0.029(12)	0.009(8)
C(5)	-0.016(3)	0.140(2)	0.336(1)	0.139(7)					
C(6A) <sup>b</sup>	-0.133(4)	0.210(2)	0.385(3)	0.147(9)					
C(6B) <sup>c</sup>	-0.044(8)	0.078(4)	0.424(4)	0.086(14)					

<sup>a</sup> Multiplicity: a = 0.5, b = 0.75, c = 0.25.

TABLE 3  
INTERATOMIC DISTANCES (Å) AND ANGLES (°)<sup>a</sup>

A. <i>Et<sub>3</sub>TeCl</i>			
Te...Cl <sup>a</sup>	3.448(4)	Cl <sup>a</sup> ...Te...Cl <sup>b</sup>	97.2(3)
Te-C(1)	2.15(3)	Te...Cl <sup>a</sup> ...Te <sup>c</sup>	82.3(3)
C(1)-C(2)	1.42(6)	Te-C(1)-C(2)	116(2)
Te...Te <sup>c</sup>	4.538(4)	C(1)-Te-C(1) <sup>d</sup>	85.8(6)
		C(1)-Te...Cl <sup>a</sup>	80.2(6)
		C(1)-Te...Cl <sup>b</sup>	165.3(6)
B. <i>Et<sub>3</sub>TeI</i>			
Te...I	3.813(5)	Te...I...Te'	79.5(4)
Te...I'	3.861(5)	Te...I...Te <sup>b</sup>	150.2(4)
Te...I''	4.494(5)	Te'...I...Te <sup>b</sup>	118.8(4)
Te-C(1)	2.16(1)	I...Te...I'	100.5(4)
Te-C(3)	2.11(1)	I...Te...I''	108.0(4)
Te-C(5)	2.16(3)	I'...Te...I''	121.4(4)
Te...C(6A)	2.92(3)	C(1)-Te-C(3)	99.0(6)
Te...Te'	4.908(5)	C(1)-Te-C(5)	96.6(8)
C(1)-C(2)	1.40(2)	C(3)-Te-C(5)	101.2(7)
C(3)-C(4)	1.46(2)	C(1)-Te...C(6)	91.8(6)
C(5)-C(6A)	1.36(3)	C(3)-Te...C(6)	80.9(7)
C(5)-C(6B)	1.29(3)	Te-C(1)-C(2)	119(1)
		Te-C(3)-C(4)	115(1)
		Te-C(5)-C(6A)	110(2)
		Te-C(5)-C(6B)	140(3)
		C-Te...I(mean)	89(2)
		C-Te...I'(mean)	168(2)

<sup>a</sup> Symmetry equivalent positions: a  $-0.5+x, 0.5-y, 0.5-z$ ; b  $0.5-x, -0.5+y, 0.5-z$ ; c  $-x, y, -z$ ; d  $z, x, y$ ; '  $-x, -y, -z$ ; ''  $0.5-x, 0.5+y, 0.5-z$ .

## Discussion

Et<sub>3</sub>TeI was prepared in a manner similar to Et<sub>3</sub>TeBr [5]; by the oxidative addition of EtI to Et<sub>2</sub>Te. However, Et<sub>3</sub>TeCl was prepared from the iodide by hydrolysis with Ag<sub>2</sub>O and subsequent neutralization with hydrochloric acid. Both the products are highly soluble in polar solvents and completely insoluble in non-polar solvents.

The structure of triethyltelluronium chloride is similar to that of the bromide [5]. Thus, in Et<sub>3</sub>TeCl the tellurium and chlorine atoms are involved in secondary interactions and alternately occupy the corners of a Te<sub>4</sub>Cl<sub>4</sub> cubane skeleton (Fig. 1). The resulting Te<sub>4</sub>Cl<sub>4</sub> unit is slightly more distorted from a regular cube than the Te<sub>4</sub>Br<sub>4</sub> skeleton in Et<sub>3</sub>TeBr. This is clear from the difference in X'TeX'' and TeXTe'' (X = Cl or Br) angles which is 14.9 in the former and 12.1° in the latter. However, both the units are more distorted than the Te<sub>4</sub>Cl<sub>4</sub> unit of Te<sub>4</sub>Cl<sub>16</sub> [9] for which the average difference in the angles is only 10.2°. The maximum deviation observed in a cubane-type compound, is 23.8° which is found in [(Et<sub>3</sub>P)CuX]<sub>4</sub> [10]. The Te...Cl distance of 3.448(4) Å in Et<sub>3</sub>TeCl is reasonably close to those reported for such

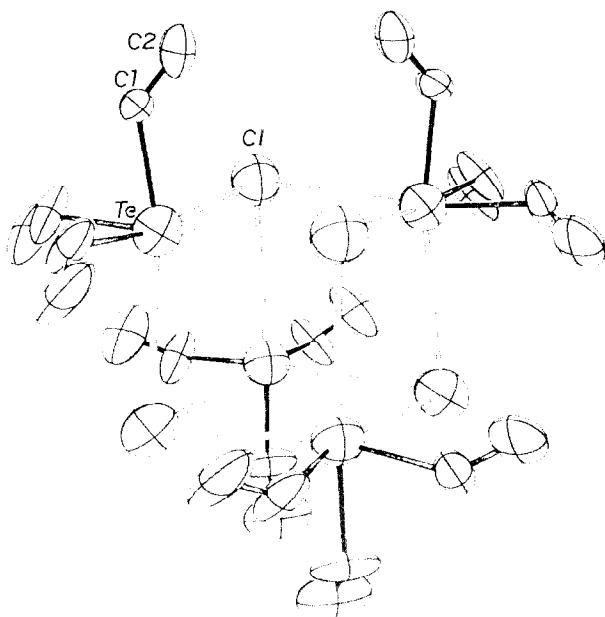


Fig. 1. ORTEP plot of the  $(\text{Et}_3\text{TeCl})_4$  molecule showing  $T_d$  symmetry. Te and Cl atoms are contoured at 50% probability. Carbon atoms are drawn arbitrarily small at the mean position of the disordered atoms. In this figure and Fig. 2 and 3, secondary interactions are shown by open bonds.

contacts in other organotellurium compounds [6,8,11–16]. The Te–C bond length of 2.15(3) Å and the CTeC angle of  $85.8(6)^\circ$  are comparable with the corresponding parameters in trimethyl and triphenyltellurium salts [1–5]. In summation, the structure of  $\text{Et}_3\text{TeCl}$  is related to that of  $\text{TeCl}_4$ , which consists of isolated cubane-type tetramers,  $\text{Te}_4\text{Cl}_{16}$ , with Te and Cl atoms occupying alternate corners of the cubane skeleton. Every Te atom has three terminal Cl atoms [9]. The substitution of these Te–Cl(t) bonds by Te–C bonds leads to the structure  $(\text{Et}_3\text{TeCl})_4$ .

Although the structures of  $\text{TeCl}_4$  and  $(\text{Et}_3\text{TeCl})_4$  are clearly related, this is not true for  $\text{TeI}_4$  and  $(\text{Et}_3\text{TeI})_4$ . The structure of  $(\text{Et}_3\text{TeI})_4$  is also quite different from the chloride and bromide. The triethyltellurium cations are associated into weakly bound centrosymmetric dimers which are bridged by iodide ions (Fig. 2). Therefore each tellurium atom is involved in only two secondary interactions and the geometry about each tellurium atom is that of a distorted square pyramid with the C(1) atom at the apical position. The rhombus  $\text{TeTeI'I'}$  is planar and the difference in  $\text{Te} \cdots \text{I} \cdots \text{Te}'$  ( $79.5(4)^\circ$ ) and  $\text{I} \cdots \text{Te} \cdots \text{I}'$  ( $100.5(4)^\circ$ ) angles is  $21.0^\circ$ , which is appreciably greater than the analogous difference in  $(\text{Et}_3\text{TeCl})_4$  and  $(\text{Et}_3\text{TeBr})_4$ . The triethyltellurium cation has a trigonal conformation of approximately  $C_3$  symmetry: the mean CTeC angle being  $97.9(7)^\circ$ . The  $\text{Te} \cdots \text{I}$  distances of 3.813(5) and 3.861(5) Å are significantly different from each other but considerably shorter than the sum of Van der Waal's radii which is 4.35 Å [17] and are of the order of the  $\text{Te} \cdots \text{I}$  secondary contacts observed in  $(\text{Me}_3\text{Te})^+(\text{MeTeI}_4)^-$  [3],  $\text{Me}_2\text{TeI}_4$  [18],  $\text{C}_{12}\text{H}_8\text{OTeI}_2$  [19],  $\text{C}_4\text{H}_8\text{OTeI}_2$  [20] and  $\text{C}_{12}\text{H}_8\text{TeI}_2$  [21]. There are no other  $\text{Te} \cdots \text{I}$  interactions less than 4.35 Å although the distance (4.494(5) Å) between Te and I' is only slightly longer. This is long for a secondary interaction but it does complete a

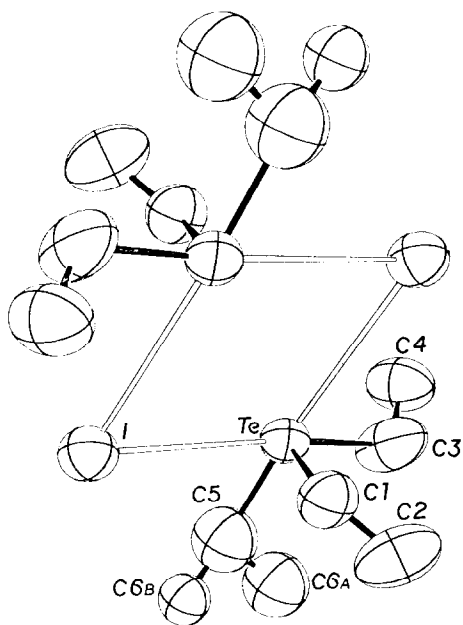


Fig. 2. ORTEP diagram of  $(\text{Et}_3\text{TeI})_2$  (50% probability ellipsoids) showing the disordered methyl group.

distorted octahedron about each tellurium atom because it is approximately *trans* to the Te–C(1) bond with the C(1)–Te–I'' angle being  $160.1(2)^\circ$ . This apparently gives rise to weakly linked sheets of dimers normal to the *a* axis as shown in Fig. 3. The structure of  $\text{Et}_3\text{TeI}$  can be related to those of  $\text{Et}_3\text{TeCl}$  or  $\text{Et}_3\text{TeBr}$  by visualizing that the cubic unit is cut into two along the *a* axis, so that each tellurium atom now still has three primary bonds to carbon atoms but only two secondary bonds to halogen atoms. This is demonstrated by the unit cell parameters (Table 1) where it can be seen that the *b* and *c* axes of  $\text{Et}_3\text{TeI}$  are similar to the cubic axes of the other two halides but the *a* axis is reduced by nearly one half when the increased size of the iodine atom is taken into account.

The structure of  $\text{Et}_3\text{TeI}$  also differs from those of  $\text{Me}_3\text{SI}$  [22] and  $\text{Me}_3\text{SeI}$  [23]. In the latter two cases, the structures are built up from isolated ion pairs with linear S or Se  $\cdots$  I linkages. However, the structure of  $\text{Et}_3\text{TeI}$  is similar to that of  $\text{Ph}_3\text{SeCl} \cdot 2\text{H}_2\text{O}$  [24] in terms of the halogen bridging even though the geometry about the selenium atom appears to be that of a distorted trigonal bipyramid.

The C–C bond lengths in both  $\text{Et}_3\text{TeI}$  and  $\text{Et}_3\text{TeCl}$  lie in the range of  $1.40(2)$ – $1.46(2)$  Å. The Te–C–C angles are in the range of  $115(2)$ – $119(2)^\circ$  with the exception of those involving C(6) as a result of the disorder problem noted in the Experimental section. These call for no comment, being entirely compatible with accepted values.

Some comparisons among the three halides are listed in Table 4. The fact that the iodide has a much lower melting point than the other two halide derivatives may reflect its dimeric as opposed to tetrameric nature in the solid state. The  $^1\text{H}$  NMR chemical shifts do show a slight trend but their similarity probably reflects the similarity of the structures in solution. Purely ionic triethyltelluronium compounds

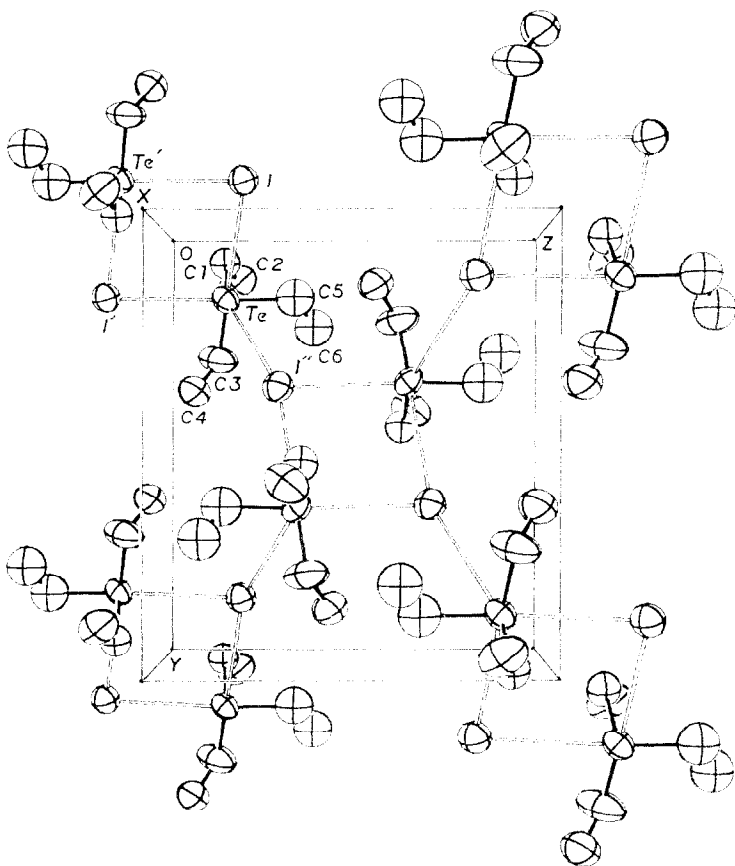


Fig. 3. Crystal packing in  $\text{Et}_3\text{TeI}$ , symmetry related positions are the same as in Table 1.

show signals in the range 2.78–2.86 ppm for the methylene proton [5] compared to 3.00–3.15 ppm in these species. The Te–C bond lengths are very similar in all three compounds but the C–Te–C bond angles open up considerably in the iodide. This

TABLE 4

COMPARISON OF SOME PROPERTIES AND PARAMETERS OF TRIETHYLTELLURONIUM HALIDES

	M.p. (°C)	$\delta(\text{CH}_2)$ (ppm)	$\delta(\text{CH}_3)$ (ppm)	Te–C (Å)	C–Te–C (°)
$\text{Et}_3\text{TeCl}$	183(d)	3.15	1.62	2.15(3)	85.8(6)
$\text{Et}_3\text{TeBr}$	155(d)	3.05	1.59	2.14(2)	89.3(6)
$\text{Et}_3\text{TeI}$	93	3.00	1.58	2.14(2) <sup>a</sup>	97.9(8) <sup>a</sup>
	Te...Te (Å)	Te...X...Te' (°)	X...Te...X' (°)	Te...X (Å)	
$\text{Et}_3\text{TeCl}$	4.538(4)	82.3(3)	97.2(3)	3.448	
$\text{Et}_3\text{TeBr}$	4.759(1)	83.8(2)	95.9(1)	3.564	
$\text{Et}_3\text{TeI}$	4.90(5)	79.5(4)	100.5(4)	3.813	

<sup>a</sup> Averaged values.



may reflect the fact that the chloride and bromide essentially have six atoms crowded around each tellurium atom while the iodide has only five. The angle  $\text{Te} \cdots \text{X} \cdots \text{Te}'$  appears to be fairly similar in all three compounds suggesting that this may be the ideal at the halide for the interactions involved in secondary bonding. The  $\text{Te} \cdots \text{X}$  distances are increasing from the chloride through to the iodide by an amount consistent with the large radius of the halogen atom. This inevitably results in the increasing  $\text{Te} \cdots \text{Te}$  distance.

### Acknowledgement

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