Benzyltriethylammonium 2,2,2,4-tetrachloro-2,5-dihydro-1,2λ^5-oxa-tellurole

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The geometry around the Te atom in the anion in $C_{13}H_{22}N^+ \cdot C_3H_3Cl_4OTe^-$ is distorted pseudo-octahedral with three Cl atoms and the O atom forming the equatorial plane, and the C atom lying opposite the tellurium lone pair. Distances and angles are: Te—O 2.0120 (18), Te—C 2.072 (2), Te—Cl 2.5239 (7), 2.5283 (7) and 2.5577 (7) Å; O—Te—C 81.61 (9), O—Te—Cl 90.69 (6), 90.99 (6) and 168.13 (5), C—Te—Cl 87.13 (8), 86.64 (8) and 86.59 (8), and Cl—Te—Cl 87.02 (2), 90.00 (3) and 173.24 (3)°. The anions are arranged in an infinite zigzag chain parallel to the *a* axis through a secondary Te···Cl bond [3.8391 (8) Å].

Comment

The addition reaction of tellurium tetrachloride to alkynes was described for the first time almost 40 years ago by Campos & Petragnani (1962). After this, only in 1979 did another report on this reaction appear in the literature (Uemura *et al.*, 1979). However, up until now, no definitive data concerning the stereochemical outcome of this reaction was provided. The addition of $TeCl_4$ to a propargylic alcohol followed by treatment with benzyltriethylammonium chloride gives rise to a compound, (I), that could be similar to that obtained by the addition reaction of trichloro(*p*-methoxyphenyl)tellurium(IV)

(I)
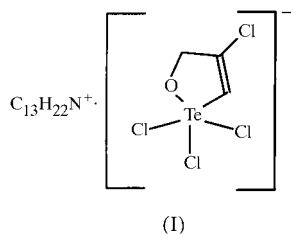
(Zukerman-Schpector *et al.*, 1999) to the corresponding propargylic alcohol. An X-ray crystal structure analysis of (I) was therefore undertaken.

The structure consists of ammonium cations and tellurate(IV) anions. The coordination around the Te atom in the $[C_3H_3Cl_4OTe]^-$ anion is distorted square pyramidal or pseudo-octahedral, with the non-bonding electron pair occupying the sixth position. The equatorial square plane composed of three Cl atoms and the O atom is slightly distorted (r.m.s. deviation of 0.046 Å), with the Te atom displaced from the plane by 0.1848 (6) Å away from the apical C1 atom. The bond lengths from the Te atom to the three surrounding Cl atoms are close to the mean value of 2.52 (3) Å calculated for 22 compounds (Allen *et al.*, 1987). The asymmetry parameter [defined as $(b2 - b1)/b1$; b's are the Te—Cl distances] of 1.74×10^{-3} and θ (Cl1—Te—Cl2 angle) of 173.24 (3)° are in good agreement with the fact that for small asymmetry, θ should be close to 180° (Landrum & Hoffmann, 1998). The Te—Cl3 distance is marginally longer than the other two Te—Cl distances; this can be explained by the fact
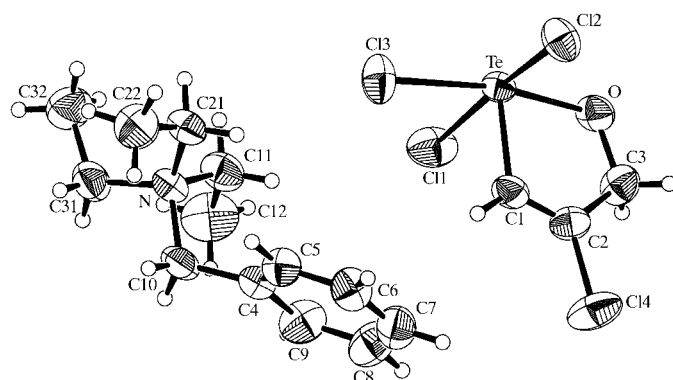

Figure 1
View of the title molecule showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of an arbitrary radius.

that the Cl3 atom is involved in a short intermolecular contact; Cl3···H6^i = 2.83, Cl3···C6^i = 3.551 (3) Å and Cl3···H6^i—C^i = 135° [symmetry code: (i) $-x, 1 - y, 1 - z$]. Whether or not this is a hydrogen bond is difficult to assertain because, as pointed out by Cotton *et al.* (1997), 'the field is getting muddier and muddier as the definition of a hydrogen bond is relaxed'. In any case, the Cl···H distance is shorter than the sum of the van der Waals radii of H and Cl (3.0 Å; Pauling, 1960). There is a secondary intermolecular bond between Te and Cl4^{ii} of 3.8391 (8) Å [symmetry code: (ii) $1 + x, y, z$], this distance being less than the sum of the van der Waals radii of Te and Cl (4.00 Å; Pauling, 1960). This secondary bond links the anions into zigzag chains parallel to **a**. The C1—Te···Cl4^{ii} angle is 144.05° rather than 180°, as might be expected if the tellurium lone pair was stereochemically inactive. The Te—C1 distance of 2.072 (2) Å is in good agreement with the sum of the Pauling (1960) single-bond covalent radii for Te (1.37 Å) and Csp^2 (0.667 Å) atoms, and with the value of 2.073 (7) Å reported by Zukerman-Schpector *et al.* (1995, and references therein). The five-membered chelate ring is planar, the r.m.s. deviation of the fitted atoms being 0.024 Å.

organic compounds

Experimental

The title compound is the addition product of the reaction between tellurium tetrachloride (0.54 g, 2.01 mmol) and propargyl alcohol (0.11 g, 2.01 mmol) in dry benzene (8 ml) at room temperature for 1 h. The crude mixture was reacted with benzyltriethylammonium chloride (0.39 g, 2.02 mmol) in dry ethanol (Petragnani *et al.*, 1976) at room temperature for 2 h. The main product of the reaction was crystallized slowly from CH_2Cl_2 as colourless crystals in 61% yield.

Crystal data

$C_{13}H_{22}N^+ \cdot C_3H_3Cl_4OTe^-$	$Z = 2$
$M_r = 516.77$	$D_x = 1.633$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.5837$ (4) Å	Cell parameters from 25
$b = 11.2598$ (5) Å	reflections
$c = 12.8491$ (7) Å	$\theta = 9.74–18.10°$
$\alpha = 94.403$ (4)°	$\mu = 1.926$ mm^{-1}
$\beta = 103.025$ (4)°	$T = 293$ (2) K
$\gamma = 98.284$ (4)°	Irregular, colourless
$V = 1051.00$ (9) Å^3	$0.30 \times 0.30 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffract-	$R_{int} = 0.012$
ometer	$\theta_{max} = 25.47°$
$\omega/2\theta$ scans	$h = -9 \rightarrow 9$
Absorption correction: ψ scan	$k = 0 \rightarrow 13$
(North *et al.*, 1968)	$l = -15 \rightarrow 15$
$T_{min} = 0.539$, $T_{max} = 0.678$	3 standard reflections
4119 measured reflections	frequency: 60 min
3906 independent reflections	intensity decay: -1.3%
3633 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.022$	$+ 0.4285P]$
$wR(F^2) = 0.059$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.068$	$(\Delta/\sigma)_{max} = 0.001$
3906 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
212 parameters	$\Delta\rho_{min} = -0.54$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0339 (10)

Table 1
Selected geometric parameters (Å, °).

Te—O	2.0120 (18)	Te—Cl2	2.5283 (7)
Te—C1	2.072 (2)	Te—Cl3	2.5577 (7)
Te—Cl1	2.5239 (7)		
O—Te—C1	81.61 (9)	Cl1—Te—Cl2	173.24 (3)
O—Te—Cl1	90.69 (6)	O—Te—Cl3	168.13 (5)
C1—Te—Cl1	87.13 (8)	C1—Te—Cl3	86.59 (8)
O—Te—Cl2	90.99 (6)	Cl1—Te—Cl3	90.00 (3)
C1—Te—Cl2	86.64 (8)	Cl2—Te—Cl3	87.02 (2)

H atoms were placed in calculated positions with fixed C—H distances (0.93 Å for Csp^2 and 0.96 Å for Csp^3), each riding on a carrier atom with an isotropic displacement parameter amounting to 1.5 (for methyl H atoms) or 1.2 (for the other H atoms) times the value of the equivalent isotropic displacement parameter of the carrier atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997), *PARST*95 (Nardelli, 1995) and *WinGX* (Farrugia, 1998).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1599). Services for accessing these data are described at the back of the journal.

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