

## Acetyldichloro[(*Z*)-2-chloro-2-phenylvinyl]tellurium(IV), helical chains of metal complexes

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The primary geometry about the Te<sup>IV</sup> atom in the title compound, [TeCl<sub>2</sub>(C<sub>8</sub>H<sub>6</sub>Cl)(C<sub>3</sub>H<sub>5</sub>O)] or C<sub>11</sub>H<sub>11</sub>Cl<sub>3</sub>OTe, is a pseudo-trigonal-bipyramidal arrangement, with two Cl atoms in apical positions, and the lone pair of electrons and C atoms in the equatorial plane. The Te<sup>IV</sup> atom is involved in three secondary interactions, two intramolecular [Te⋯O = 2.842 (3) Å and Te⋯Cl3 = 3.209 (1) Å] and one intermolecular [Te⋯Cl = 3.637 (1) Å], the latter giving rise to a helical chain. These helices are linked by C—H⋯O interchain interactions.

### Comment

Organyltellurium trichlorides are quite insoluble in the solvents usually used for NMR studies. On the other hand, the corresponding diorganotellurium dichlorides are quite soluble, especially in chloroform. For this reason, the organylytellurium trichloride obtained from the reaction of TeCl<sub>4</sub> with phenylethyne was treated with an excess of acetone, giving the corresponding  $\alpha$ -tellurium acetone dichloride, which was recrystallized from methylene dichloride to give the title compound, (I). In order to overcome the problem that the NMR analysis gives no indication of the stereochemistry of the double bond, an X-ray analysis of (I) was undertaken.

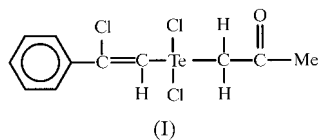


Fig. 1 shows that the addition reaction proceeds in a *syn* fashion to give the *Z* isomer only. The coordination around the Te<sup>IV</sup> atom is consistent with a pseudo-trigonal-bipyramidal bond configuration, with two Cl atoms occupying the axial

positions, and C atoms and the lone pair of electrons occupying the equatorial positions. This configuration is in complete agreement with the valence-shell electron-pair repulsion (VSEPR) model (Gillespie, 1972). The average quadruple angle,  $\alpha_4^E$ , defined as  $[(360 - \text{Cl}-\text{Te}-\text{Cl}) + (360 - \text{C}-\text{Te}-\text{C})]/4$ , for the Te lone pair of electrons, is 112.6°, a value typical for TeX<sub>4</sub>E configurations (Zukerman-Schpector *et al.*, 1996, and references therein). The Te—Cl axial lengths are 0.13 and 0.16 Å longer than the sum of the normal covalent radii (2.36 Å; Ziolo & Troup, 1983) and may be compared with the values found in related compounds (Zukerman-Schpector *et al.*, 1995, 1996). The Te—C bond distances of 2.077 (4) and 2.130 (4) Å (Table 1) are slightly different than the predicted values of 2.037 and 2.142 Å obtained using Pauling's radii for Te (1.37 Å), Csp<sup>2</sup> (0.667 Å) and Csp<sup>3</sup> (0.772 Å) atoms (Pauling, 1960).

From the Te⋯C2 distance of 2.987 (4) Å and the carbon non-bonded radius of 1.25 Å (O'Keefe & Hyde, 1981), the non-bonded radius for the Te atom may be estimated to be 1.74 Å, a value close to that found in related compounds (Zukerman-Schpector *et al.*, 1995, 1996, and references therein). The equatorial plane formed by atoms C1, C9, Cl3

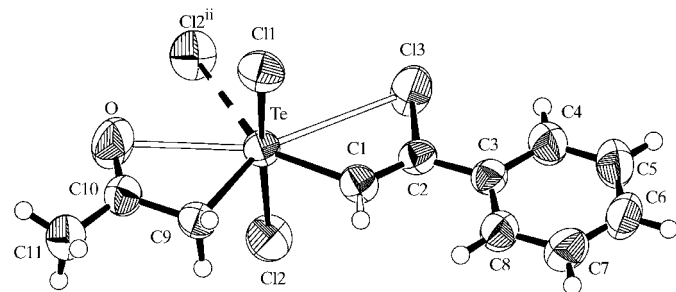


Figure 1

The molecular structure of (I) showing the atom labelling and 50% probability displacement ellipsoids. The intramolecular secondary bond is shown as an unfilled bond and the intermolecular interactions as dashed lines [symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ].

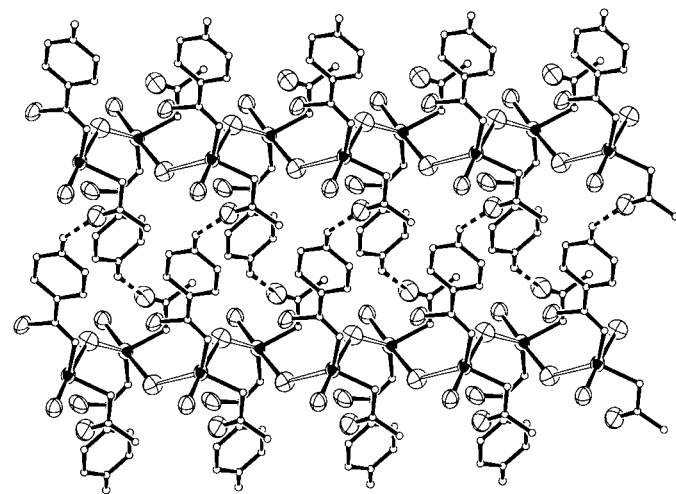


Figure 2

The packing of the molecules showing two helices. The Te⋯Cl secondary bonds are shown as unfilled bonds and the linking C—H⋯O interactions are shown as dashed lines.

and Cl2<sup>i</sup> [symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ] is slightly distorted (r.m.s. deviation 0.0757 Å) and the Te atom is 0.261 (2) Å out of this plane towards the Cl1 atom. The phenyl ring makes a dihedral angle of 10.7 (2)° with the C1/C9/Cl3/Cl2<sup>i</sup> equatorial plane.

The Te atom makes three non-bonded interactions, two intramolecular [Te···O = 2.842 (3) Å and Te···Cl3 = 3.209 (1) Å] and one intermolecular [Te···Cl2<sup>i</sup> = 3.637 (1) Å], which are significantly shorter than the sum of the van der Waals radii (3.60 and 4.01 Å for Te···O and Te···Cl, respectively; Pauling, 1960). The last interaction links the molecules in a spiral fashion. These adjacent helices are extended into a three-dimensional supramolecular array by a C6—H6···O(*x* - 1, *y*, *z*) interchain interaction, as shown in Fig. 2 and Table 2, where several C—H···Cl interactions, corresponding to H···Cl contacts shorter than Pauling's van der Waals radii sum (3.01 Å; Pauling, 1960), are also listed.

Taking into account the secondary interactions of tellurium, the real coordination number increases to seven. In order to decide which would be the best description of the polyhedron, the following dihedral angles were calculated: C1, Cl1, C9/Cl1, C9, O = 46.9 (1)°, Cl1, Cl3, Cl2<sup>i</sup>/Cl1, O, Cl2<sup>i</sup> = 41.3 (1)° and C9, Cl2, Cl2<sup>i</sup>/C9, Cl2<sup>i</sup>, O = 33.3 (2)°. Thus, following Kouba and Wreford (1976), the arrangement about the Te atom should be described as a distorted monocapped octahedron, with the O atom capping the Cl1/C9/Cl2<sup>i</sup> face.

## Experimental

Suitable crystals of (I) were obtained by slow evaporation from a methylene dichloride at 277 K.

### Crystal data

C <sub>11</sub> H <sub>11</sub> Cl <sub>3</sub> OTe	<i>D<sub>x</sub></i> = 1.859 Mg m <sup>-3</sup>
<i>M<sub>r</sub></i> = 393.15	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>n</i>	Cell parameters from 25 reflections
<i>a</i> = 12.661 (1) Å	<i>θ</i> = 10.2–18.4°
<i>b</i> = 6.0330 (8) Å	<i>μ</i> = 2.67 mm <sup>-1</sup>
<i>c</i> = 18.4940 (2) Å	<i>T</i> = 293 (2) K
<i>β</i> = 96.19 (1)°	Irregular, yellow
<i>V</i> = 1404.4 (3) Å <sup>3</sup>	0.40 × 0.25 × 0.20 mm
<i>Z</i> = 4	

### Data collection

Enraf–Nonius CAD-4 diffractometer	<i>R</i> <sub>int</sub> = 0.011
<i>ω</i> - <i>2θ</i> scans	<i>θ</i> <sub>max</sub> = 26.0°
Absorption correction: <i>ψ</i> scan (North <i>et al.</i> , 1968)	<i>h</i> = 0 → 15
<i>T</i> <sub>min</sub> = 0.394, <i>T</i> <sub>max</sub> = 0.587	<i>k</i> = 0 → 7
2894 measured reflections	<i>l</i> = -22 → 22
2765 independent reflections	3 standard reflections
2195 reflections with <i>I</i> > 2σ( <i>I</i> )	frequency: 30 min
	intensity decay: 0.8%

**Table 1**

Selected geometric parameters (Å, °).

Te—C1	2.077 (4)	Te—Cl2	2.4918 (12)
Te—C9	2.130 (4)	Te—Cl1	2.5158 (11)
C1—Te—C9	98.32 (14)	C1—Te—Cl1	86.47 (11)
C1—Te—Cl2	87.16 (11)	C9—Te—Cl1	87.26 (13)
C9—Te—Cl2	88.03 (13)	Cl2—Te—Cl1	171.46 (4)

### Refinement

Refinement on <i>F</i> <sup>2</sup>	<i>w</i> = 1/[σ <sup>2</sup> ( <i>F<sub>o</sub></i> <sup>2</sup> ) + (0.0332 <i>P</i> ) <sup>2</sup> + 0.9575 <i>P</i> ]
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.025	where <i>P</i> = ( <i>F<sub>o</sub></i> <sup>2</sup> + 2 <i>F<sub>c</sub></i> <sup>2</sup> )/3
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.071	(Δ/ <i>σ</i> ) <sub>max</sub> < 0.001
<i>S</i> = 1.04	Δ <i>ρ</i> <sub>max</sub> = 0.52 e Å <sup>-3</sup>
2765 reflections	Δ <i>ρ</i> <sub>min</sub> = -0.48 e Å <sup>-3</sup>
146 parameters	H-atom parameters constrained

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C6—H6···O <sup>i</sup>	0.93	2.57	3.360 (6)	144
C4—H4···Cl3	0.93	2.56	2.990 (4)	109
C1—H1···Cl1 <sup>ii</sup>	0.93	2.77	3.626 (4)	154
C11—H11C···Cl2 <sup>iii</sup>	0.96	2.87	3.674 (6)	142

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) -*x*, -*y*, -*z*; (iii)  $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$ .

H atoms were located on stereochemical grounds and were refined riding on their carrier atoms [C—H = 0.93–0.97 Å; *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C) for methyl H atoms or 1.2*U*<sub>eq</sub>(C) for all others].

Data collection and cell refinement: *CAD-4 Software* (Enraf–Nonius, 1989); data reduction: *XCAD4* (Harms & Wocadlo, 1995); structure solution: *SIR92* (Altomare *et al.*, 1993); structure refinement: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *PARST95* (Nardelli, 1995), *PLATON* (Spek, 1998) and *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1512). Services for accessing these data are described at the back of the journal.

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