Crystal data

$C_{12}H_{10}N_3O^+ \cdot H_2PO_4^- \cdot H_2O$
$M_r = 327.23$
Triclinic
$P\bar{1}$
$a = 7.503$ (8) Å
$b = 9.702$ (5) Å
$c = 10.501$ (3) Å
$\alpha = 93.93$ (4)°
$\beta = 103.99$ (3)°
$\gamma = 107.26$ (7)°
$V = 700.1$ (8) Å^3
$Z = 2$
$D_x = 1.552$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 41 reflections
$\theta = 4.90$–12.74°
$\mu = 0.232$ mm^{-1}
$T = 293$ (2) K
Block
0.40 × 0.30 × 0.30 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
3084 measured reflections
2467 independent reflections
2177 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

$\theta_{\max} = 25°$
$h = -1 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$
3 standard reflections every 97 reflections intensity decay: 5.17%

Refinement

Refinement on F^2
$R(F) = 0.033$
$wR(F^2) = 0.089$
$S = 1.086$
2467 reflections
256 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.2386P]$ where $P = F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.076 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

P1—O2	1.497 (2)	N1—C5	1.351 (2)
P1—O4	1.5318 (14)	N2—C6	1.344 (2)
P1—O1	1.551 (2)	N2—C7	1.381 (2)
P1—O3	1.552 (2)	N3—C6	1.337 (2)
O5—N1	1.332 (2)	N3—C12	1.381 (2)
N1—C1	1.344 (2)		
O2—P1—O4	114.30 (9)	O5—N1—C1	119.9 (2)
O2—P1—O1	108.69 (10)	O5—N1—C5	118.9 (2)
O4—P1—O1	107.70 (9)	C1—N1—C5	121.2 (2)
O2—P1—O3	111.32 (10)	N1—C1—C2	120.4 (2)
O4—P1—O3	106.16 (10)	C1—C2—C3	120.1 (2)
O1—P1—O3	108.46 (10)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O5	0.89 (4)	1.67 (4)	2.563 (3)	176 (3)
N2—H2N···O1*W*	0.83 (2)	1.89 (3)	2.717 (3)	173 (2)
O3—H3O···O2$^{\rm i}$	0.77 (3)	1.80 (3)	2.565 (2)	173 (3)
O1*W*—H1*WA*···O5$^{\rm ii}$	0.83 (3)	1.97 (3)	2.800 (2)	174 (3)
O1*W*—H1*WB*···O2$^{\rm iii}$	0.83 (3)	1.91 (3)	2.742 (3)	175 (4)
N3—H4O···O4$^{\rm iv}$	0.87 (3)	1.69 (3)	2.555 (2)	168 (3)

Symmetry codes: (i) $1 - x, 2 - y, 2 - z$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $-x, 1 - y, 1 - z$; (iv) $x, y - 1, z$.

Refined C—H distances are in the range 0.90 (2)–0.98 (2) Å.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1261). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1930–1932

Acetonyldichloro[(*Z*)-2-chloro-1-methyl-2-phenylethenyl]tellurium(IV)

J. Zukerman-Schpector,[a,b]* Robinson L. Camillo,[a] João V. Comasseto,[b] Rodrigo L. O. R. Cunha,[b] Francisco C. D. Lemos[a] and I. Caracelli[a]

[a]*Laboratório de Cristalografia, Estereodinâmica e Modelagem Molecular, Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13565-905 São Carlos, SP, Brazil, and* [b]*Instituto de Química, Universidade de São Paulo, São Paulo, Brazil. E-mail: julio@power.ufscar.br*

(*Received 11 June 1999; accepted 6 July 1999*)

Abstract

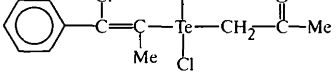
The primary geometry about the $\rm Te^{IV}$ atom in the title compound, $C_{12}H_{13}Cl_3OTe$ or $[TeCl_2(C_9H_8Cl)(C_3H_5O)]$, is a pseudo-trigonal-bipyramidal arrangement with the

Acta Crystallographica Section C
ISSN 0108-2701 © 1999

lone pair of electrons and the C atoms occupying the equatorial positions, and the Cl atoms occupying the axial positions. There are three secondary interactions, two intramolecular [Te···O 2.926 (4) and Te···Cl 3.180 (1) Å] and one intermolecular [Te···Cl 3.659 (1) Å], which link the molecules. Significant distances and angles are: Te—Cl 2.4870 (13) and 2.5096 (12), Te—C 2.119 (4) and 2.122 (4) Å, Cl—Te—Cl 171.78 (5), Cl—Te—C 88.07 (11), 87.68 (15), 86.28 (11) and 87.17 (15), and C—Te—C 97.59 (17)°.

Comment

Organyltellurium trichlorides are 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 organyltellurium trichloride obtained from the reaction of $TeCl_4$ with 1-phenyl-1-propyne was treated with an excess of acetone giving the corresponding α-tellurium acetone dichloride, which, in turn, was recrystallized from methylene dichloride to give the title compound, (I). As the NMR analysis give no indication of the stereochemistry of the compound, an X-ray crystal structure analysis was undertaken.



$C_6H_5\text{—}C(Cl)\text{=}C(Me)\text{—}TeCl_2\text{—}CH_2\text{—}C(\text{=}O)\text{—}Me$

(I)

As can be seen from Fig. 1, the *Z* isomer was obtained. The Te^{IV} atom presents a primary pseudo-trigonal-bipyramidal configuration formed by four bonds to the ligands (two Cl and two C atoms) and one lone pair of electrons, which, together with the two C atoms, occupy equatorial positions, while the two Cl atoms occupy the axial positions. This configuration is in complete agreement with the valence-shell electron-pair repulsion (VSEPR) model (Gillespie, 1972). The average quadruple angle, α_4^E, defined as [(360 − Cl—Te—Cl) + (360 − C—Te—C)]/4, for the Te lone pair of electrons is 112.7°, a value typical for TeX_4E configurations (Zukerman-Schpector *et al.*, 1996, and references therein). The Te—Cl bond lengths of 2.5096 (12) and 2.4870 (13) Å are in good agreement, within experimental accuracy, with the values found in other $TeCl_2RR'$ compounds (Zukerman-Schpector *et al.*, 1995, and references therein); as expected for trigonal-bipyramidal coordination, the axial bonds are 0.15 and 0.13 Å longer than the sum of the normal covalent radii (2.36 Å; Ziolo & Troup, 1983). The Te—C bond distances of 2.119 (4) and 2.122 (4) Å are longer than the predicted value of 2.037 Å obtained using Pauling's radii for Te (1.37 Å) and Csp^2 (0.667 Å) atoms (Pauling, 1960).

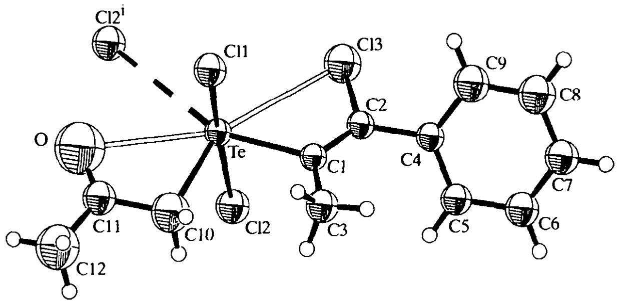


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. The Te intramolecular secondary interactions are shown as open bonds. [Symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

From the Te···C2 distance of 2.970 (4) Å and the C non-bonded radius of 1.25 Å (O'Keefe & Hyde, 1981), the non-bonded radius of the Te atom may be estimated to be 1.72 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, C10, Cl3 and Cl2^i [symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$] is slightly distorted (r.m.s. deviation 0.0576 Å) and the Te atom is 0.217 (2) Å out of this plane towards the Cl1 atom. The phenyl ring is planar within experimental accuracy [σ_{av}, defined as $(\Sigma_i d_i^2/N-3)^{1/2}$, is 0.0069 Å, where *d* is the deviation of the atoms from the best least-squares plane through them and *N* is the number of atoms included in the plane] and makes a dihedral angle of 54.1 (1)° with the C1/C10/Cl3/Cl2^i equatorial plane.

There are three secondary interactions, two intramolecular [Te···O 2.926 (4) and Te···Cl3 3.180 (1) Å] and one intermolecular [Te···Cl2^i 3.659 (1) Å]. This last interaction links the molecules in a spiral fashion. Taking into account these three secondary interactions, the Te atom is seven-coordinated. In order to decide which would be the best description of the polyhedron, the following dihedral angles were calculated: C1–Cl1–C10/Cl1–C10–O 42.0 (2), Cl1–Cl3–Cl2^i/Cl1–O–Cl2^i 48.2 (1) and C10–Cl2–Cl2^i/C10–Cl2^i–O 29.4 (2)°. Thus, the arrangement about the Te atom, following Kouba & Wreford (1976), should be described as a distorted monocapped octahedron, with the O atom capping the Cl1/C10/Cl2^i face.

Experimental

The organyltellurium trichloride obtained from the reaction of $TeCl_4$ with 1-phenyl-1-propyne was treated with an excess of acetone giving the corresponding α-tellurium acetone dichloride. This was recrystallized from methylene dichloride to give the title compound.

Crystal data

$C_{12}H_{13}Cl_3OTe$
$M_r = 407.17$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 12.269$ (2) Å
$b = 6.081$ (1) Å
$c = 20.176$ (2) Å
$\beta = 102.629$ (9)°
$V = 1468.9$ (4) Å^3
$Z = 4$
$D_x = 1.841$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 9.56$–18.12°
$\mu = 2.552$ mm^{-1}
$T = 293$ (2) K
Irregular
0.35 × 0.20 × 0.20 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.469$, $T_{\max} = 0.629$
2807 measured reflections
2726 independent reflections

2387 reflections with $F^2 > 2\sigma F^2$
$R_{\rm int} = 0.015$
$\theta_{\max} = 25.46°$
$h = -14 \rightarrow 14$
$k = -7 \rightarrow 0$
$l = 0 \rightarrow 24$
3 standard reflections
frequency: 30 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.086$
$S = 1.047$
2726 reflections
156 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 2.6277P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.797$ e Å^{-3}
$\Delta\rho_{\min} = -0.844$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Te—C1	2.119 (4)	Te—Cl2	2.4870 (13)
Te—C10	2.122 (4)	Te—Cl1	2.5096 (12)
C1—Te—C10	97.59 (17)	C1—Te—Cl1	86.28 (11)
C1—Te—Cl2	88.07 (11)	C10—Te—Cl1	87.17 (15)
C10—Te—Cl2	87.68 (15)	Cl2—Te—Cl1	171.78 (5)

H atoms were placed in calculated positions with fixed C—H distances (0.93 Å for C*sp*^{2} and 0.96 Å for C*sp*^{3}), each riding on their carrier atoms with an isotropic displacement parameter of 1.5 (for methyl H atoms) or 1.2 (for the other H atoms) times the equivalent isotropic displacement parameter of the attached atom. The highest peak and the deepest hole in the final difference Fourier map are 0.90 and 0.88 Å, respectively, from the Te atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL*97.

This work has received partial support from FAPESP (Proc. 94/1213-5), CNPq and CAPES. RLC thanks FAPESP for a fellowship (98/05787-7). The X-ray facility at the Instituto de Química–USP was established with the assistance of FAPESP (94/2061-4).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1570). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1932–1935

Tetrakis(cyclopropylammonium) cyclotetraphosphate tetrahydrate

El Hassane Soumhi,[a] Ismael Saadoune,[a] Ahmed Driss[b] and Tahar Jouini[b]

[a]*Département de Chimie, FST Marrakech, BP 618 Marrakech, Morocco, and* [b]*Département de Chimie, Faculté des Sciences, 1060 Campus Universitaire, Tunis, Tunisia.*
E-mail: fstg@cybernet.net.ma

(*Received 27 October 1998; accepted 29 June 1999*)

Abstract

In the structure of the title phosphate, $4C_3H_8N^+\cdot P_4O_{12}^{4-}\cdot$-$4H_2O$, the $(P_4O_{12})^{4-}$ cyclic anions are linked through OH$\cdots$O—P hydrogen bonds to build layers parallel to the *ab* plane. These layers are themselves interconnected by means of N—H$\cdots$O hydrogen bonds originating from the $(C_3H_5NH_3)^+$ cations.

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Acta Crystallographica Section C
ISSN 0108-2701 © 1999