C1	0.3805 (2)	−0.0484 (2)	0.1701 (2)	0.0351 (6)
C2	0.4758 (2)	−0.0388 (2)	0.2422 (2)	0.0367 (6)
C3	0.5523 (2)	0.0257 (3)	0.2287 (2)	0.0451 (8)
C4	0.6393 (2)	0.0425 (3)	0.2944 (2)	0.0588 (9)
C5	0.6513 (3)	−0.0054 (4)	0.3749 (3)	0.0721 (12)
C6	0.5766 (3)	−0.0701 (4)	0.3897 (3)	0.0769 (13)
C7	0.4896 (3)	−0.0873 (3)	0.3239 (3)	0.0577 (10)
C8	0.3199 (2)	−0.1381 (3)	0.1726 (2)	0.0455 (8)
N2	0.1668 (2)	0.1566 (2)	0.0506 (2)	0.0422 (6)
C9	0.2129 (3)	0.2297 (3)	0.1110 (2)	0.0613 (10)
C10	0.1627 (3)	0.3008 (3)	0.1506 (3)	0.0725 (12)
C11	0.0605 (3)	0.2988 (3)	0.1268 (3)	0.0660 (11)
C12	0.0116 (3)	0.2262 (3)	0.0642 (2)	0.0573 (9)
C13	0.0671 (2)	0.1575 (3)	0.0287 (2)	0.0483 (8)

Table 2. *Selected geometric parameters* (Å, °)

Zn1—O1	2.039 (2)	O3—N1	1.262 (3)
Zn1—N2	2.114 (2)	N1—C8	1.364 (4)
Zn1—O3	2.152 (2)	C1—C8	1.385 (4)
O1—C1	1.254 (3)	C1—C2	1.496 (4)
O2—N1	1.242 (3)		
O1^i—Zn1—O1	169.86 (11)	C1—O1—Zn1	129.9 (2)
O1—Zn1—N2^i	90.85 (8)	N1—O3—Zn1	128.6 (2)
O1—Zn1—N2	95.92 (9)	O2—N1—O3	117.9 (3)
N2^i—Zn1—N2	96.30 (13)	O2—N1—C8	119.4 (3)
O1^i—Zn1—O3	89.33 (8)	O3—N1—C8	122.7 (3)
O1—Zn1—O3	83.23 (8)	O1—C1—C8	126.2 (3)
N2^i—Zn1—O3	172.26 (9)	O1—C1—C2	116.4 (3)
N2—Zn1—O3	89.28 (9)	C8—C1—C2	117.3 (3)
O3—Zn1—O3^i	85.68 (13)	N1—C8—C1	126.1 (3)

Symmetry code: (i) $\frac{1}{2} - x, y, -z$.

Upon being harvested from the mother liquid, crystals of the title compound were colorless with well defined faces. After several days in a sealed vial, they began to display a flat white crust, indicating that they had undergone slow decomposition, perhaps by loss of the coordinated pyridine. For X-ray data collection, a small colorless crystal was cut from a sample with evidence of decomposition at the surface. The crystal was covered with a thin layer of epoxy and did not show signs of significant change during the course of the data collection. Three intensity standards changed by less than 1.5% in the 28 h span during which data were gathered.

The empirical absorption correction was based on nine complete ψ scans. The crystal was indexed using a body-centered monoclinic lattice in which the *a* and *c* axes were the shortest repeats in the *ac* plane. The principal lattice repeats were verified by oscillation photographs about *a*, *b*, *c* and the body diagonal [111].

The development and refinement of the structure proceeded routinely. A direct-methods calculation (Sheldrick, 1990) located all of the non-H atoms. All H atoms were located in a difference Fourier map and were refined independently. Anisotropic displacement parameters were used for all non-H atoms.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4-PC*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bonamico, M., Dessy, G., Fares, V. & Scaramuzza, L. (1972). *J. Chem. Soc. Dalton Trans.* pp. 2477–2483.
Enraf–Nonius (1993). *CAD-4-PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Dichloro[(Z)-2-chloro-2-phenylvinyl]-(4-methoxyphenyl)tellurium(IV)

JULIO ZUKERMAN-SCHPECTOR

Universidade Federal de São Carlos, Departamento de Química, Caixa Postal 676, 13565-905 São Carlos SP, Brazil

JOÃO VALDIR COMASSETO AND HELIO ANDRÉ STEFANI

Instituto de Química, Universidade de São Paulo, 01498 São Paulo SP, Brazil

Abstract

In the title compound, [TeCl$_2$(C$_8$H$_6$Cl)(C$_7$H$_7$O)], the Te^{IV} atom is in a trigonal bipyramidal configuration with the lone pair of electrons occupying one of the equatorial positions. Distances and angles are: Te—Cl 2.521 (2) and 2.485 (2), Te—C 2.073 (7) and 2.110 (7) Å (aryl) ; Cl—Te—Cl 117.65 (8), Cl—Te—C 87.1 (2), 90.6 (2), 90.8 (2) and 90.6 (2), C—Te—C 95.0 (3)°.

Comment

Vinylic tellurides are intermediates in the synthesis of vinyllithium compounds (Barros, Comasseto & Berriel, 1989), which are, in turn, important intermediates in organic synthesis, either as precursors of the widely used vinylcuprate compounds or as nucleophiles leading to chain elongation products by reaction with many electrophiles (Comasseto & Berriel, 1990; Lipshutz, 1989). The crystal structure determination of compound (1) was

undertaken because a knowledge of its stereochemistry is required to predict successive reaction pathways; it is supposed that transformations occur with retention of the olefin geometry.

(1)
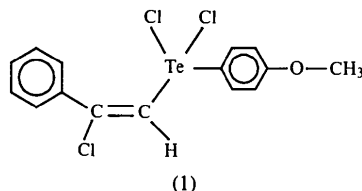
The Te^{IV} atom presents a typical trigonal bipyramidal configuration formed from four bonds to the ligands (two Cl and two C atoms) and one lone pair of electrons, which, together with atoms C(1) and C(9), occupy equatorial sites, while the Cl atoms occupy axial positions. This configuration is in complete agreement with the valence-shell electron-pair-repulsion model (VSEPR) (Gillespie, 1972). The quadruple average angle of the lone pair, a_4^E, is 110.6° and is a typical value found in the TeX$_4$E configurations (Hargittai & Rozsondai, 1986). As expected for trigonal-bipyramidal coordination, the axial bonds are 0.16 and 0.13 Å longer than the sum of the normal covalent radii (2.36 Å; Ziolo & Troup, 1983). On the other hand, the C—Te—C angle of 95.0° is smaller than the average value of 99° found in these species.

Both phenyl rings are planar within experimental accuracy [σ_{av} defined as $(\Sigma_i d_i^2/N{-}3)^{1/2}$ is 0.02 for both rings] and the dihedral angle between the planes is 68.9 (3)°.

The C(1)···C(3) distance of 2.51 (1) Å is in good agreement with the value of 2.50 Å predicted from the 1···3 non-bonded radius for C atoms of 1.25 Å (O'Keeffe & Hyde, 1981). From the Te···C(2) distance of 3.030 (8) Å the non-bonded radius for the Te may be estimated to be 1.78 Å, which is in good agreement with the values found in related compounds (Zukerman-Schpector, Castellano, Oliva, Comasseto & Stefani, 1991, and references therein).

The molecules are linked through C—H···Cl interactions: Cl(2)···C(11^i) = 3.666 (8), Cl(2)···H(C11^i) =

2.755 (8) Å, Cl(2)···H(C11^i)—C(11^i) = 140.5 (8)°; Cl(1)···C(4^{ii}) = 3.850 (8), Cl(1)—H(C4^{ii}) = 2.717 (8) Å, Cl(1)···H(C4^{ii})—C(4^{ii}) = 171.7 (8)° [symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, z$].

Experimental

Compound (1) was synthesized as reported by Comasseto, Stefani, Chieffi & Zukerman-Schpector (1991). Crystals were obtained from CHCl$_3$/petroleum ether solution.

Crystal data

[TeCl$_2$(C$_8$H$_6$Cl)(C$_7$H$_7$O)]	Mo $K\alpha$ radiation
M_r = 443.23	λ = 0.71073 Å
Orthorhombic	Cell parameters from 25
$Pbca$	reflections
a = 19.076 (2) Å	θ = 9–18°
b = 8.348 (1) Å	μ = 2.30 mm^{-1}
c = 20.608 (2) Å	T = 292 K
V = 3282 (1) Å^3	Irregular
Z = 8	0.30 × 0.25 × 0.15 mm
D_x = 1.794 Mg m^{-3}	Yellowish

Data collection

Enraf–Nonius CAD-4 diffractometer	1460 observed reflections [$I > 3\sigma(I)$]
$\omega/2\theta$ scans	R_{int} = 0.017
Absorption correction:	θ_{max} = 25°
refined from ΔF	h = −1 → 22
(*DIFABS*; Walker & Stuart, 1983)	k = 0 → 9
T_{min} = 0.51, T_{max} = 0.70	l = 0 → 24
2558 measured reflections	2 standard reflections
2404 independent reflections	frequency: 30 min
	intensity decay: 0.6%

Refinement

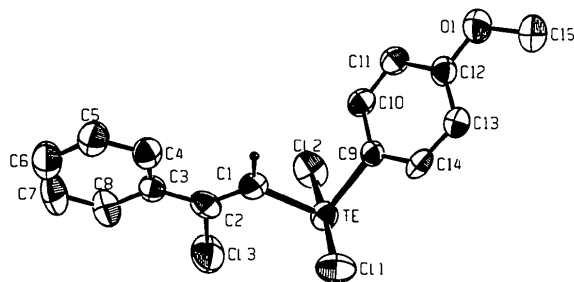|	
Refinement on F	$w = 1/[\sigma^2(|F_o|$
R = 0.0335	$+ 0.00005|F_o|^2]$
wR = 0.0302	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.25	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
1460 reflections	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
182 parameters	Atomic scattering factors
H atoms refined with one overall U_{iso} [0.079 (7) Å^2]	from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Fig. 1. The molecular structure of the title complex showing the atom-labelling scheme and 50% displacement ellipsoids.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}a_i.a_j.$$

	x	y	z	B_{eq}
Te	0.3226 (1)	0.3676 (1)	0.3493 (1)	3.97 (2)
Cl(1)	0.2050 (1)	0.2866 (3)	0.3945 (1)	5.49 (9)
Cl(2)	0.4407 (1)	0.4446 (3)	0.3091 (1)	6.2 (1)
Cl(3)	0.3975 (2)	0.4683 (3)	0.4857 (1)	7.6 (1)
O	0.3081 (3)	−0.1131 (7)	0.1191 (2)	5.0 (2)
C(1)	0.3670 (4)	0.2144 (9)	0.4170 (4)	4.1 (3)
C(2)	0.3959 (4)	0.2645 (9)	0.4729 (4)	4.0 (3)
C(3)	0.4213 (4)	0.160 (1)	0.5259 (3)	3.9 (3)
C(4)	0.4101 (4)	−0.004 (1)	0.5224 (4)	4.1 (3)
C(5)	0.4309 (4)	−0.101 (1)	0.5736 (4)	5.0 (4)
C(6)	0.4621 (5)	−0.036 (1)	0.6264 (4)	5.5 (4)
C(7)	0.4760 (5)	0.125 (1)	0.6292 (4)	6.7 (4)
C(8)	0.4543 (5)	0.224 (1)	0.5795 (4)	5.3 (4)

C(9)	0.3140 (4)	0.1925 (8)	0.2759 (3)	3.2 (3)
C(10)	0.3723 (4)	0.110 (1)	0.2540 (4)	4.2 (3)
C(11)	0.3695 (4)	0.0064 (9)	0.2030 (4)	4.0 (3)
C(12)	0.3051 (4)	−0.0118 (9)	0.1712 (3)	3.7 (3)
C(13)	0.2459 (4)	0.0654 (9)	0.1923 (4)	3.9 (3)
C(14)	0.2505 (4)	0.1673 (9)	0.2451 (3)	3.7 (3)
C(15)	0.2474 (5)	−0.126 (1)	0.0801 (4)	6.3 (4)

Table 2. *Selected geometric parameters (Å, °)*

Te—Cl(1)	2.521 (1)	Te—Cl(2)	2.485 (2)
Te—C(1)	2.073 (7)	Te—C(9)	2.110 (7)
C(2)—Cl(3)	1.722 (8)	O—C(12)	1.368 (9)
O—C(15)	1.41 (1)	C(1)—C(2)	1.34 (1)
C(2)—C(3)	1.48 (1)	C(3)—C(4)	1.39 (1)
C(3)—C(8)	1.38 (1)	C(4)—C(5)	1.39 (1)
C(5)—C(6)	1.35 (1)	C(6)—C(7)	1.37 (2)
C(7)—C(8)	1.38 (1)	C(9)—C(10)	1.38 (1)
C(9)—C(14)	1.38 (1)	C(10)—C(11)	1.36 (1)
C(11)—C(12)	1.40 (1)	C(12)—C(13)	1.37 (1)
C(13)—C(14)	1.38 (1)		
Cl(1)—Te—Cl(2)	177.65 (8)	C(3)—C(4)—C(5)	119.5 (7)
Cl(1)—Te—C(1)	87.1 (2)	C(4)—C(5)—C(6)	120.2 (8)
Cl(1)—Te—C(9)	90.6 (2)	C(5)—C(6)—C(7)	120.8 (9)
Cl(2)—Te—C(1)	90.8 (2)	C(6)—C(7)—C(8)	119.9 (9)
Cl(2)—Te—C(9)	90.6 (2)	C(3)—C(8)—C(7)	120.0 (8)
C(1)—Te—C(9)	95.0 (3)	C(10)—C(9)—C(14)	118.6 (7)
C(12)—O—C(15)	117.3 (6)	C(9)—C(10)—C(11)	122.4 (7)
Cl(3)—C(2)—C(1)	116.5 (6)	C(10)—C(11)—C(12)	117.7 (7)
Cl(3)—C(2)—C(3)	117.6 (6)	O—C(12)—C(11)	113.4 (7)
C(1)—C(2)—C(3)	125.7 (7)	O—C(12)—C(13)	125.0 (7)
C(2)—C(3)—C(4)	119.5 (7)	C(11)—C(12)—C(13)	121.5 (7)
C(2)—C(3)—C(8)	120.8 (7)	C(12)—C(13)—C(14)	119.1 (7)
C(4)—C(3)—C(8)	119.6 (7)	C(9)—C(14)—C(13)	120.6 (7)

Data were corrected for Lp effects. The structure was solved by direct methods. H atoms were included as fixed contributors at positions found in a difference synthesis and refined with one overall isotropic temperature factor which converged to 0.079 (7) Å². The refinement was by full-matrix least-squares methods.

Programs used: *SHELXS*86 (Sheldrick, 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

References

Barros, S. M., Comasseto, J. V. & Berriel, J. (1989). *Tetrahedron Lett.* **30**, 7353–7356.

Comasseto, J. V. & Berriel, J. (1990). *Synth. Commun.* **20**, 1681–1682.

Comasseto, J. V., Stefani, H. A., Chieffi, A. & Zukerman-Schpector, J. (1991). *Organometallics*, **10**, 845–846.

Gillespie, R. J. (1972). In *Molecular Geometry*. London: Van Nostrand Reinhold.

Hargittai, I. & Rozsondai, B. (1986). *The Chemistry of Organic Selenium and Tellurium Compounds*, Vol. 1, edited by S. Patai & Z. Rappoport, pp. 63–155. New York: John Wiley.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Lipshutz, B. H. Ed. (1989). *Tetrahedron*, **45**, 349–578.

O'Keefe, M. & Hyde, B. G. (1981). *Structure and Bonding in Crystals*, Vol. I, edited by M. O'Keefe & A. Navrotsky, pp. 227–254. New York: Academic Press.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELX86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Ziolo, R. F. & Troup, J. M. (1983). *J. Am. Chem. Soc.* **105**, 229–235.

Zukerman-Schpector, J., Castellano, E. E., Oliva, G., Comasseto, J. V. & Stefani, H. A. (1991). *Acta Cryst.* C**47**, 960–962.

Bis[bis(3,5-dimethyl-1-pyrazolyl)phosphinato]copper(II), [Cu{O₂P(N₂C₃HMe₂)₂}₂]

Susan Folkert, Clinton D. Bryan and A. Wallace Cordes

Department of Chemistry and Biochemistry, University of Arkansas, Fayetteville, AR 72701, USA

Paulmony Tharmaraj and Vadapalli Chandrasekhar

Department of Chemistry, Indian Institute of Technology, Kanpur 208 016, India

Abstract

The title compound, [Cu(C₁₀H₁₄N₄O₂P)₂], consists of pairs of bis(dimethylpyrazolyl)phosphinato groups coordinated to copper(II) atoms sitting on inversion centers. The compound has Cu—N bond distances of 2.009 (4) and 2.010 (4) Å and an N—Cu—N angle of 89.27 (13)°. The phosphinato O atoms weakly coordinate the copper ions with bond distances of 2.490 (3) Å.

Comment

The title compound, (I), was obtained as a hydrolysis product in a study of the ligation behavior of tris(3,5-dimethyl-1-pyrazolyl)phosphine oxide.

(I)
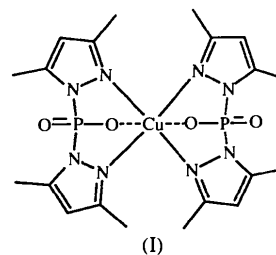
The ready conversion of the phosphine oxide to the phosphinato ion has been noted before: the ligation of tris(3,5-dimethyl-1-pyrazolyl)phosphine oxide with an