Refinement

Refinement on F^2
$R(F) = 0.0562$
$wR(F^2) = 0.1387$
$S = 1.110$
2500 reflections
201 parameters
H atoms riding, with one common $U = 0.085$ (3) Å^2
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.5960P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.338$ e Å^{-3}
$\Delta\rho_{\min} = -0.233$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.050 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
F	0.5492 (2)	0.0574 (4)	0.10106 (12)	0.1100 (9)
O1	0.6346 (2)	−0.0005 (4)	0.57096 (13)	0.0799 (8)
O2	0.8216 (2)	0.1232 (3)	0.38575 (13)	0.0706 (6)
N1	0.7531 (2)	0.0640 (3)	0.49662 (14)	0.0522 (6)
N2	0.5782 (2)	0.0575 (3)	0.42271 (14)	0.0553 (6)
C1	0.8854 (2)	0.2551 (4)	0.6084 (2)	0.0485 (7)
C2	0.9683 (2)	0.3531 (5)	0.5941 (2)	0.0642 (8)
C3	0.9966 (3)	0.5317 (5)	0.6298 (2)	0.0777 (10)
C4	0.9420 (3)	0.6100 (5)	0.6808 (2)	0.0773 (10)
C5	0.8589 (3)	0.5118 (6)	0.6947 (2)	0.0844 (11)
C6	0.8309 (3)	0.3358 (5)	0.6587 (2)	0.0706 (9)
C7	0.8539 (2)	0.0612 (4)	0.5709 (2)	0.0582 (7)
C8	0.6524 (2)	0.0364 (4)	0.5038 (2)	0.0557 (7)
C9	0.6284 (2)	0.0928 (4)	0.3609 (2)	0.0492 (7)
C10	0.7456 (2)	0.0976 (4)	0.4114 (2)	0.0512 (7)
C11	0.5902 (2)	0.1134 (4)	0.2748 (2)	0.0522 (7)
C12	0.4797 (2)	0.1054 (4)	0.2147 (2)	0.0503 (7)
C13	0.3872 (2)	0.1248 (4)	0.2381 (2)	0.0584 (7)
C14	0.2851 (2)	0.1156 (5)	0.1769 (2)	0.0675 (8)
C15	0.2720 (3)	0.0865 (5)	0.0902 (2)	0.0681 (8)
C16	0.3614 (3)	0.0681 (5)	0.0640 (2)	0.0710 (9)
C17	0.4615 (2)	0.0771 (4)	0.1266 (2)	0.0622 (8)

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

F—C17	1.351 (3)	N2—C8	1.362 (3)
O1—C8	1.216 (3)	N2—C9	1.392 (3)
O2—C10	1.214 (3)	C1—C7	1.503 (4)
N1—C10	1.374 (3)	C9—C11	1.327 (4)
N1—C8	1.375 (3)	C9—C10	1.483 (4)
N1—C7	1.465 (3)	C11—C12	1.455 (4)
C10—N1—C8	111.0 (2)	C11—C9—C10	122.7 (2)
C10—N1—C7	125.1 (2)	N2—C9—C10	104.8 (2)
C8—N1—C7	123.8 (2)	O2—C10—N1	125.3 (3)
C8—N2—C9	111.1 (2)	O2—C10—C9	129.1 (3)
N1—C7—C1	111.9 (2)	N1—C10—C9	105.6 (2)
O1—C8—N2	127.0 (2)	C9—C11—C12	130.3 (2)
O1—C8—N1	125.4 (3)	F—C17—C16	118.1 (3)
N2—C8—N1	107.5 (2)	F—C17—C12	117.3 (3)
C11—C9—N2	132.5 (2)	C16—C17—C12	124.6 (3)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995).

This work has received partial support from FAPESP (Proc. 94/1213-5), CNPq, FAPEAL, CAPES and FINEP.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1146). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(*Z*)-1-(Dichloro-*p*-methoxyphenyltelluro)-1-phenyl-2-thiophenylethene

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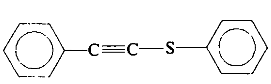
Abstract

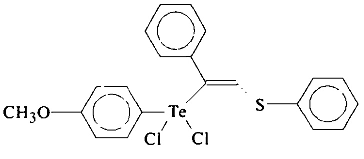
The Te^{IV} atom in the title compound, $C_{21}H_{18}Cl_2OSTe$, is in a trigonal bipyramidal configuration with the lone pair of electrons occupying one of the equatorial positions. Distances and angles are: Te—Cl 2.5140 (10) and 2.5012 (10), Te—C 2.127 (3) and 2.121 (4) Å (aryl); Cl—Te—Cl 173.07 (4), Cl—Te—C 90.96 (10), 87.71 (9), 90.09 (10) and 85.36 (9), C—Te—C 99.92 (13)°.

Comment

Addition of sodium organyltellurolate anions to terminal acetylenes containing a conjugated aromatic (Barros, Dabdoub, Dabdoub & Comasseto, 1989) or unsaturated (Dabdoub, Dabdoub & Comasseto, 1992; Dabdoub & Dabdoub, 1995) group occurs regio- and stereospecifically, giving disubstituted vinylic tellurides with a *Z* configuration. Following our continuing interest in the synthesis of trisubstituted vinylic tellurides, the hydrotelluration reaction of 1-phenyl-2-thiophenyl acetylene, (1), was carried out. Analysis of the reaction product showed that only one isomer was obtained. As its regio- and stereochemistry could not be determined by the usual spectroscopic methods (^{1}H NMR, ^{13}C NMR, IR), the liquid product was transformed into the solid telluro derivative, (2), by reaction with thionyl chloride in petroleum ether, and a crystal structure determination was undertaken.



(1)



(2)

The coordination around the Te^{IV} atom is consistent with a trigonal bipyramidal bond configuration formed by four bonds to two Cl and two C atoms of the ligands and one lone pair of electrons which, together with atoms C1 and C15, occupy equatorial sites while the Cl atoms occupy axial positions. This configuration is in complete agreement with the valence-shell electron-pair repulsion (VSPER) model (Gillespie, 1972). The average quadrupole angle α_4^E for the Te lone pair of electrons is 111.8°, a value typical for TeX_4E configurations (Hargittai & Rozsnodai, 1986; Zukerman-Schpector, Comasseto & Stefani, 1995, and references therein). The Te—Cl bond lengths of 2.5140 (10) and 2.5012 (10) Å and the Cl—Te—Cl angle of 173.07 (4)° are in good agreement, to within experimental accuracy, with the values found in other TeCl$_2$*RR'* compounds (Zukerman-Schpector *et al.*, 1995, and references therein); as expected for trigonal bipyramidal coordinations, the axial bonds are 0.15 and 0.14 Å longer than the sum of the normal covalent radii (2.36 Å; Ziolo & Troup, 1983). The C—Te—C angle of 99.92 (13)° is less than the ideal value of 120°; the distortion has been explained on the basis that the lone-pair–bond-pair repulsions are greater than bond-pair–bond-pair repulsions (Chadha, Drake & Kahn, 1983).

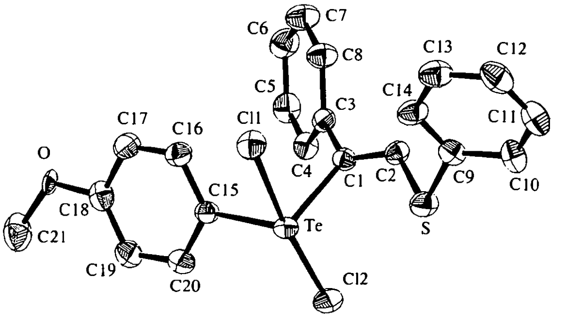


Fig. 1. The molecular structure of $C_{21}H_{18}Cl_2OSTe$ showing the atom labelling. 50% probability displacement ellipsoids are shown.

The C2···C3 distance of 2.480 (5) 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'Keefe & Hyde, 1981). From the Te···C2 distance of 2.948 (4) Å, the non-bonded radius for the Te atom may be estimated to be 1.70 Å, marginally shorter than the values found in related compounds (Zukerman-Schpector *et al.*, 1995, and references therein).

The phenyl rings are planar within experimental accuracy, σ_{av} [defined as $(\Sigma_i d_i^2/N - 3)^{1/2}$] being 3.5×10^{-3}, 7.2×10^{-4} and 4.6×10^{-3} for C3–C8 (ring *A*), C9–C14 (ring *B*) and C15–C20 (ring *C*), respectively; the dihedral angles between the rings are A^B 62.3 (2), A^C 35.8 (2) and B^C 35.6 (2)°. The molecules are linked through C—H···Cl interactions [Cl1···C11^{i} = 3.764 (5), Cl1···H11^{i} = 2.86 Å, Cl1···H11^{i}—C11^{i} = 140°; Cl2···C17^{ii} = 3.759 (4), Cl2···H17^{ii} = 2.84 Å, Cl2···H17^{ii}—C17^{ii} = 152°; symmetry codes: (i) $x - 1, y, z$; (ii) $-x, y - \frac{1}{2}, \frac{3}{2} - z$].

The specificity of the hydrotelluration of the 1-phenyl-2-thiophenyl acetylene, confirmed here, and the importance of both vinylic tellurides (Barros *et al.*, 1989; Dabdoub *et al.*, 1992; Dabdoub & Dabdoub, 1995; Dabdoub, Cassol & Barbosa, 1996) and vinylic sulfides (Trost & Lavoie, 1983, and references therein; Luh & Ni, 1990) as useful synthetic intermediates prompted us to extend our study to other thioalkynes (Dabdoub, Dabdoub, Pereira, Barbosa & Zukerman-Schpector, 1996).

Experimental

Crystals were obtained by slow evaporation from petroleum ether.

Crystal data

$C_{21}H_{18}Cl_2OSTe$	Mo $K\alpha$ radiation
M_r = 516.939	λ = 0.71073 Å

Monoclinic
$P2_1/c$
$a = 9.944\ (1)$ Å
$b = 13.171\ (1)$ Å
$c = 15.920\ (2)$ Å
$\beta = 97.480\ (10)°$
$V = 2067.3\ (4)$ Å^3
$Z = 4$
$D_x = 1.6609$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 9.30–18.84°$
$\mu = 1.806$ mm^{-1}
$T = 293$ K
Irregular
0.35 × 0.20 × 0.20 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3787 measured reflections
3642 independent reflections
2951 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0216$
$\theta_{\rm max} = 25°$
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 0$
$l = 0 \rightarrow 18$
2 standard reflections
frequency: 30 min
intensity decay: 1.1%

Refinement

Refinement on F^2
$R(F) = 0.0261$
$wR(F^2) = 0.0610$
$S = 1.082$
3642 reflections
236 parameters
H atoms fixed with one common U
$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2 + 1.0896P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.303$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.421$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Te	0.26679 (2)	−0.05169 (2)	0.84215 (1)	0.03332 (9)
Cl1	0.23243 (11)	0.11760 (8)	0.90900 (6)	0.0491 (2)
Cl2	0.31633 (11)	−0.20873 (7)	0.76344 (7)	0.0526 (3)
S	0.59201 (10)	−0.01052 (8)	0.87855 (7)	0.0470 (3)
O	−0.3169 (3)	−0.0235 (2)	0.6450 (2)	0.0550 (8)
C1	0.3780 (4)	0.0308 (3)	0.7593 (2)	0.0323 (8)
C2	0.5071 (4)	0.0473 (3)	0.7879 (2)	0.0362 (8)
C3	0.3083 (3)	0.0704 (3)	0.6782 (2)	0.0331 (8)
C4	0.2406 (4)	0.0069 (3)	0.6171 (2)	0.0394 (9)
C5	0.1767 (4)	0.0461 (3)	0.5423 (2)	0.0475 (10)
C6	0.1773 (4)	0.1487 (3)	0.5276 (3)	0.0527 (11)
C7	0.2458 (5)	0.2124 (3)	0.5873 (3)	0.0582 (12)
C8	0.3103 (4)	0.1735 (3)	0.6617 (3)	0.0476 (10)
C9	0.6836 (4)	0.0939 (3)	0.9280 (2)	0.0404 (9)
C10	0.8240 (4)	0.0881 (3)	0.9418 (3)	0.0505 (10)
C11	0.8957 (4)	0.1664 (4)	0.9854 (3)	0.0611 (12)
C12	0.8293 (5)	0.2488 (4)	1.0128 (3)	0.0611 (12)
C13	0.6901 (5)	0.2546 (3)	0.9990 (3)	0.0557 (11)
C14	0.6168 (4)	0.1765 (3)	0.9564 (3)	0.0496 (10)
C15	0.0696 (4)	−0.0427 (3)	0.7734 (2)	0.0350 (8)
C16	0.0097 (4)	0.0506 (3)	0.7502 (2)	0.0414 (9)
C17	−0.1188 (4)	0.0534 (3)	0.7075 (3)	0.0448 (9)
C18	−0.1905 (4)	−0.0361 (3)	0.6883 (2)	0.0417 (9)
C19	−0.1330 (4)	−0.1282 (3)	0.7122 (3)	0.0491 (10)
C20	−0.0027 (4)	−0.1312 (3)	0.7550 (3)	0.0460 (10)
C21	−0.4011 (4)	−0.1106 (4)	0.6284 (3)	0.0692 (14)

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

Te—C15	2.121 (4)	S—C9	1.776 (4)
Te—C1	2.127 (3)	O—C18	1.363 (5)
Te—Cl2	2.5012 (10)	O—C21	1.426 (5)
Te—Cl1	2.5140 (10)	C1—C2	1.323 (5)
S—C2	1.749 (4)	C1—C3	1.479 (5)
C15—Te—C1	99.92 (13)	C2—S—C9	101.1 (2)
C15—Te—Cl2	90.96 (10)	C18—O—C21	118.4 (3)
C1—Te—Cl2	87.71 (9)	C2—C1—C3	124.4 (3)
C15—Te—Cl1	90.09 (10)	C2—C1—Te	115.5 (3)
C1—Te—Cl1	85.36 (9)	C3—C1—Te	120.0 (2)
Cl2—Te—Cl1	173.07 (4)	C1—C2—S	123.7 (3)

The H atoms were found in difference Fourier maps and included as fixed contributors with an overall isotropic displacement parameter that converged to $U = 0.069\ (3)$ Å^2.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995).

This work has received partial support from FAPESP (Proc. 94/1213–5), CNPq, CAPES and FINEP.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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