$S = 1.091$
2684 reflections
286 parameters
H-atom parameters
constrained

$(\Delta/\sigma)_{max} = 0.015$
$\Delta\rho_{max} = 0.368$ e Å^{-3}
$\Delta\rho_{min} = -0.245$ e Å^{-3}
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

O1—CO′—N1—C1A	−169.7 (2)
C1—O1—CO′—N1	−169.5 (2)
CO′—N1—C1A—C1′	−64.0 (3)
N1—C1A—C1′—N2	−25.2 (3)
C1A—C1′—N2—C2A	177.6 (2)
C1′—N2—C2A—C2′	−60.1 (3)
N2—C2A—C2B—C2G	177.9 (2)
C2A—C2B—C2G—C2D1	66.3 (5)
C2A—C2B—C2G—C2D2	−168.0 (4)
N2—C2A—C2′—N3	−40.2 (3)
C2A—C2′—N3—C3A	−173.4 (2)
C2′—N3—C3A—C3′	53.8 (4)
N3—C3A—C3′—O4	38.3 (4)
C3A—C3′—O4—C4M	179.1 (2)
N3—C3A—C3B1—C3G1	175.5 (3)
N3—C3A—C3B2—C3G2	−61.8 (5)

Three of the cyclooctane ring atoms (C3D1, C3D2 and C3E) showed positional disorder, a feature not unexpected in an eight-membered ring. This was modelled in terms of two unequal sites of occupancies 0.6 and 0.4. The disordered atoms were allowed to refine with isotropic displacement parameters (Mukhopadhyyay *et al.*, 1998). All the H atoms (except the disordered atoms) were geometrically fixed and allowed to ride on the corresponding non-H atoms. Two of the cyclooctane ring distances were restrained.

Data collection: *P3 Software* (Siemens, 1991). Cell refinement: *P3 Software*. 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: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1983, 1995).

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Trichloro[(*Z*)-2-chloro-1,2-diphenylvinyl]-tellurium(IV)

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Abstract

In the title structure, [Te(C$_{14}$H$_{10}$Cl)Cl$_3$] or C$_{14}$H$_{10}$Cl$_4$Te, the Te atom is in a pseudo-trigonal bipyramidal configuration, with the lone pair of electrons, a vinyl-C atom and a Cl atom occupying the equatorial positions and the other two Cl atoms occupying the axial positions. Distances and angles are: Te—Cl 2.280 (2), 2.463 (5) and 2.469 (5), and Te—C 2.132 (7) Å; Cl—Te—Cl 174.15 (12), 87.9 (2) and 89.4 (2), and Cl—Te—C 86.8 (5), 88.4 (5) and 97.8 (2)°.

Comment

Vinylic tellurides are emerging as important synthetic reagents (Comasseto *et al.*, 1997). The most popular method of preparing such reagents consists of the addition of nucleophilic tellurium to acetylenes (Comasseto, 1993). The addition of electrophilic tellurium to acetylenes as a method of accessing vinylic tellurides has been studied much less (Comasseto *et al.*, 1997). Recently, we developed a method of preparing tri- and tetrasubstituted vinylic tellurides by means of the addi-

tion of tellurium tetrachloride to acetylenes followed by reduction of the vinyltellurium trichloride with sodium borohydride and alkylation of the intermediate vinylic tellurate (Chieffi *et al.*, 1997). In order to overcome the problem concerning the determination of the stereochemistry of the double bond in the products by routine methods, an X-ray analysis of the title compound, (I), was undertaken.
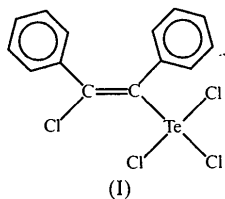
(I)

Fig. 1 shows that the addition reaction proceeds in a *syn* fashion to give the Z adduct. The coordination around the Te^{IV} atom is consistent with a trigonal bipyramidal bond configuration, with two Cl atoms occupying axial positions while the C atom, one Cl atom and the lone pair of electrons occupy the equatorial positions. This configuration is in complete agreement with the valence shell electron-pair repulsion model (VSEPR; Gillespie, 1972). The average quadruple angle, α_4^E, for the Te lone pair of electrons is 112.03°, a value typical for TeX_4E configurations (Hargittai & Rozsnodai, 1986; Zukerman-Schpector *et al.*, 1995, and references therein). The Te—Cl bond distance of 2.132 (7) Å is longer than the value of 2.037 Å predicted using the Pauling (1960) radii for Te (1.37 Å) and Csp^2 (0.667 Å). The Te—Cl1 and Te—Cl2 axial lengths are 0.11 and 0.10 Å longer, respectively, 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 (Chakravorty *et al.*, 1985).

The molecules in the crystal are associated *via* C—H···π interactions between C4—H4 and the C9–C14 phenyl ring. Following the ideas of Ciunik *et al.* (1998), this kind of interaction should be characterized
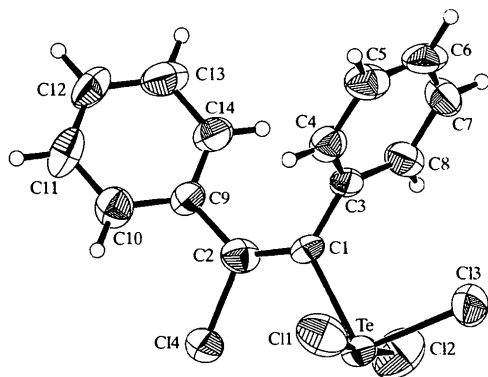
by three parameters. In the present structure these are the H4···Cg^i (Cg is the centroid of the C9–C14 ring) distance of 2.978 Å, the C4—H4···Cg^i angle of 155° and the angle between the H···Cg vector and the plane of the aromatic ring, which should be calculated as the H4···Cg^i···C10^i angle, of 94° [symmetry code: (i) $\frac{1}{2} - x$, y, $\frac{1}{2} + z$]. These values are in the expected ranges of 2.7–3.4 Å, 140–160° and 80–100°, respectively, as described by Ciunik *et al.* (1998).

There is a secondary intramolecular bond between Te and Cl4 [2.996 (3) Å]. This distance is less than the sum of the van der Waals radii (Pauling, 1960) of Te and Cl (4.00 Å).

Experimental

A mixture of tellurium tetrachloride (1.35 g, 0.005 mol) and diphenylacetylene (1.00 g, 0.0055 mol) was refluxed in 20 ml carbon tetrachloride for 2 h. The resulting yellow solution was evaporated to leave a yellow oil from which crystals of (I) formed (yield: 1.70 g, 77%). Two recrystallizations from glacial acetic acid furnished yellow prisms (m.p. 398–401 K).

Crystal data

$C_{14}H_{10}Cl_4Te$	Mo $K\alpha$ radiation
$M_r = 447.62$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
$Pca2_1$	reflections
$a = 21.031 (3)$ Å	$\theta = 9.96–17.67°$
$b = 8.2010 (8)$ Å	$\mu = 2.527$ mm^{-1}
$c = 9.215 (1)$ Å	$T = 293 (2)$ K
$V = 1589.4 (3)$ Å^3	Irregular
$Z = 4$	$0.35 \times 0.20 \times 0.15$ mm
$D_x = 1.871$ Mg m^{-3}	Brown
D_m not measured	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.043$
diffractometer	$\theta_{max} = 25°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 24$
Absorption correction:	$k = -9 \rightarrow 9$
ψ scan (North *et al.*,	$l = 0 \rightarrow 10$
1968)	3 standard reflections
$T_{min} = 0.472$, $T_{max} = 0.703$	frequency: 30 min
2873 measured reflections	intensity decay: 0.9%
1495 independent reflections	
1058 reflections with	
$F^2 > 2\sigma F^2$	

Refinement

|	
Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$\Delta\rho_{max} = 0.379$ e Å^{-3}
$wR(F^2) = 0.069$	$\Delta\rho_{min} = -0.485$ e Å^{-3}
$S = 1.090$	Extinction correction: none
1495 reflections	Scattering factors from
148 parameters	*International Tables for*
H atoms: see below	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Fig. 1. A 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 spheres of arbitrary radius.

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

Te—C1	2.132 (7)	Te—C11	2.469 (5)
Te—C13	2.280 (2)	C14—C2	1.763 (8)
Te—C12	2.463 (5)	C1—C2	1.307 (10)
C1—Te—C13	97.8 (2)	C1—Te—C11	86.8 (5)
C1—Te—C12	88.4 (5)	C13—Te—C11	87.9 (2)
C13—Te—C12	89.4 (2)	C12—Te—C11	174.15 (12)

The phenyl rings were refined as rigid groups with their H atoms placed in calculated positions at fixed C—H distances of 0.93 Å, each riding on a carrier atom, and with isotropic displacement parameters amounting to 1.2 times the value of the equivalent isotropic displacement parameter of the atom to which they were attached. The Flack (1983) parameter is 0.02 (5).

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.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1044). Services for accessing these data are described at the back of the journal.

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1,3a,5-Triphenyl-3a,4,5,6-tetrahydro-1,2,4-oxadiazolo[4,5-a][1,5]benzodiazepine

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Abstract

The title compound, $C_{28}H_{23}N_3O$, has a *cis* ring-fusion tricyclic structure, which is formed from a benzene ring, a seven-membered heterocyclic diazepine ring and a 1,2,4-oxadiazole ring. The 1,5-diazepine ring has a slightly distorted boat-like conformation, whereas the 1,2,4-oxadiazole ring adopts an envelope conformation.

Comment

Benzodiazepine derivatives containing additional rings are potential pharmaceutical agents (Sternbach, 1979; Xu & Jin, 1994; Bock *et al.*, 1989). In connection with investigations on possible approaches to novel benzodiazepine derivatives with additional fused heterocyclic rings (Xu & Jin, 1992, 1999; Xu *et al.*, 1998), we have tested the C=N double bond of the 2,3-dihydro-1*H*-1,5-benzodiazepines as a dipolarophile in the 1,3-dipolar cycloaddition of nitrile oxides (Xu *et al.*, 1999) and obtained some 3a,4,5,6-tetrahydro-1,2,4-oxadiazolo[4,5-*a*][1,5]benzodiazepine derivatives with potential central nervous system depressant and anti-HIV activities. In order to elucidate the conformation of the 1,3-cycloadduct of 2,3-dihydro-1*H*-1,5-benzodiazepine with nitrile oxide, the crystal structure of the title compound, (I), was determined.

(I)
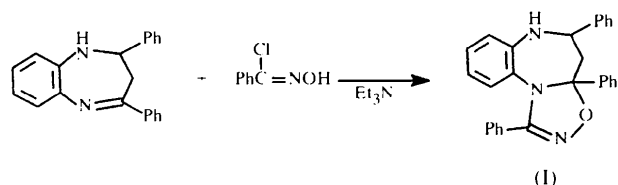
The molecular backbone of (I) is a tricyclic system, formed from a benzene ring, a seven-membered heterocyclic 1,5-diazepine ring and a 1,2,4-oxadiazole ring. The central seven-membered heterocycle is in a slightly distorted boat-like conformation and is *cis*-fused to the 1,2,4-oxadiazole ring at N2 and C9, while the latter moiety is in an envelope conformation. The phenyl group on C7 is equatorial to the central ring, but that on C9 is axial (Fig. 1), hence the cycloaddition reaction is a *cis*-addition reaction. The molecule contains two chiral