Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2931 measured reflections
2630 independent reflections
2586 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\rm max} = 74.92°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = -21 \rightarrow 21$
6 standard reflections
frequency: 166.7 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.020$
2630 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.2478P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.034$
$\Delta\rho_{\rm max} = 0.162$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.153$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0084 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C2	1.3325 (16)	N8—C3*A*	1.3796 (15)
N1—N8	1.3624 (15)	C6—C7	1.3698 (18)
N4—C5	1.3272 (16)	C6—C5	1.4273 (17)
N4—C3*A*	1.3432 (17)	N3—C3*A*	1.3346 (16)
N8—C7	1.3719 (15)	N3—C2	1.3523 (18)
C2—N1—N8	101.09 (10)	N3—C3*A*—N4	127.95 (11)
C5—N4—C3*A*	116.39 (11)	N3—C3*A*—N8	109.24 (11)
N1—N8—C7	127.37 (10)	N4—C3*A*—N8	122.78 (11)
N1—N8—C3*A*	110.18 (9)	N4—C5—C6	122.65 (11)
C7—N8—C3*A*	122.36 (10)	C6—C7—N8	115.18 (11)
C7—C6—C5	120.60 (11)	N1—C2—N3	117.03 (12)
C3*A*—N3—C2	102.45 (10)		

Only reflections which had $F^2 \geq 3\sigma(F^2)$ were used in the refinement. The refinement included both positional and isotropic displacement parameters for the H atoms; the C—H range = 0.96 (2)–1.02 (2) Å. The value of the Flack (1983) parameter [−0.2 (3); 996 Friedel-related reflections] was unreliable.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *DREADD* (Blessing, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1048). Services for accessing these data are described at the back of the journal.

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Dichloro[(*E*)-2-chloro-1-(2-hydroxyprop-2-yl)vinyl](4-methoxyphenyl)tellurium(IV)

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Abstract

The geometry around the Te$^{\rm IV}$ atom in the title compound, $C_{12}H_{15}Cl_3O_2Te$ or $[TeCl_2(C_5H_8ClO)(C_7H_7O)]$, is pseudo-trigonal bipyramidal, with the Cl atoms in the apical positions and the Te lone pair occupying the fifth position in the equatorial plane. By including the intermolecular secondary Te···Cl contact [3.485 (1) Å] *trans* to the Te—C$_{\rm vinyl}$ bond, the structure may be regarded as pseudo-octahedral. The molecules are associated *via* an O—H···Cl hydrogen bond to form centrosymmetric dimeric units, which in turn are arranged in an infinite zigzag chain along the *b* axis through the secondary Te···Cl bond. Distances and angles are: Te—Cl 2.4820 (8) and 2.5600 (8), Te—C

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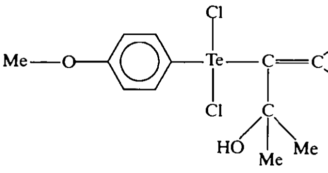
2.124 (3) and 2.129 (3) Å; Cl—Te—Cl 173.24 (3), Cl—Te—C 92.42 (8), 92.86 (8), 87.06 (8) and 88.03 (8), and C—Te—C 98.02 (12)°.

Comment

The result of the addition of aryltellurium trichlorides to alkynes depends on the structure of the substrate. Addition to terminal arylalkynes gives (*Z*)-1-telluro-1-chloroethenes (Comasseto *et al.*, 1991), whereas addition to 3-hydroxyalkynes gives mixtures of (*Z*)- and (*E*)-telluroalkenes in variable ratios (Zeni *et al.*, 1999). Mixtures of regioisomers are formed and the regiochemistry of the products is also dependent on the structure of the propargyl alcohol. To understand the regio- and stereochemical outcome of this reaction, the structures of the products formed are being determined.

In the current experiment, the *E* isomer, (I), was produced in excess over the *Z* isomer (approximate ratio 80:20 *via* NMR), and the more concentrated isomer crystallized first and was the only solid isolated from the mixture. In the structure of the *E* isomer, the geometry around the Te^{IV} atom can be described as pseudo-octahedral, consisting of two C atoms of the ligands, two primary bonded chloride ligands, one secondary Te···Cl interaction and the lone pair of electrons occupying the fourth equatorial site (Singh *et al.*, 1990). The secondary Te···Cl interaction is approximately *trans* to C_{vinyl} [$Te\cdots Cl1^{i}$ = 3.485 (1) Å and $C1—Te\cdots Cl1^{i}$ = 166.38 (9)°; symmetry code: (i) $-x, -y, -z$], the distance being less than the sum of the van der Waals radii of Te and Cl (4.00 Å; Pauling, 1960). The C—Te—C angle of 98.02 (12)° could be expected to be closer to 90° were the long Te···Cl intermolecular interaction slightly stronger.



(I)

The molecules are associated into dimers by means of a centrosymmetric O—H···Cl hydrogen bond: $O1\cdots Cl2^{ii}$ 3.252 (3), $H\cdots Cl2^{ii}$ 2.44 Å and $O1—H\cdots Cl2^{ii}$ 170.0° [symmetry code: (ii) $-x, 1-y, -z$]. Although the coordination about Te appears to be pseudo-octahedral, in each monomer unit, the Te^{IV} atom adopts the characteristic four-coordinated geometry based on a pseudo-trigonal–bipyramidal polyhedron, formed by four bonds to two Cl and two C atoms of the ligands, and one lone pair of electrons which, together with the C atoms, 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, α_4^E, defined as the mean of the four angles made by the lone pair, is 112.2° (ignoring the long Te···Cl interaction), a value typical for TeX_4E configurations (Hargittai & Rozsnodai, 1986; Zukerman-Schpector *et al.*, 1995, and references therein). The Te—C bond distances of 2.124 (3) and 2.129 (3) Å are longer than predicted (2.04 Å) using Pauling's radius for Te (1.37 Å) and Csp^2 (0.67 Å) (Pauling, 1960). It can be postulated that this is due to the Te···O1 intramolecular interaction of 2.693 (3) Å, which is shorter than the sum of the van der Waals radii of 3.60 Å (Pauling, 1960). The Te—Cl axial lengths are 0.20 and 0.12 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 other $TeCl_2RR'$-type compounds (Dabdoub *et al.*, 1990; Zukerman-Schpector *et al.*, 1995; Singh *et al.*, 1990). This elongation can be ascribed to the fact that one Cl atom is involved in a hydrogen bond while the other forms a secondary bond to the Te atom of a neighboring molecule.

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 for 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 phenyl ring is planar within experimental accuracy [σ_{av} defined as $(\Sigma_i d_i^2/N-3)^{1/2}$ is 0.014 Å], making a dihedral angle of 82.1 (3)° with the C3—C1—C2 moiety.

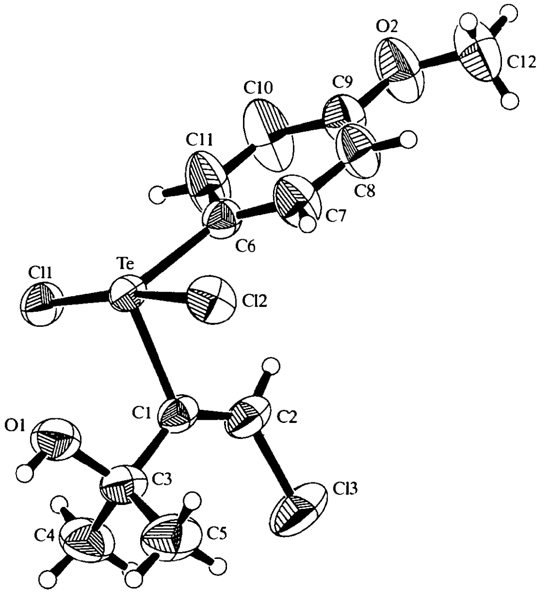


Fig. 1. View of the title molecule showing the labeling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of an arbitrary radius.

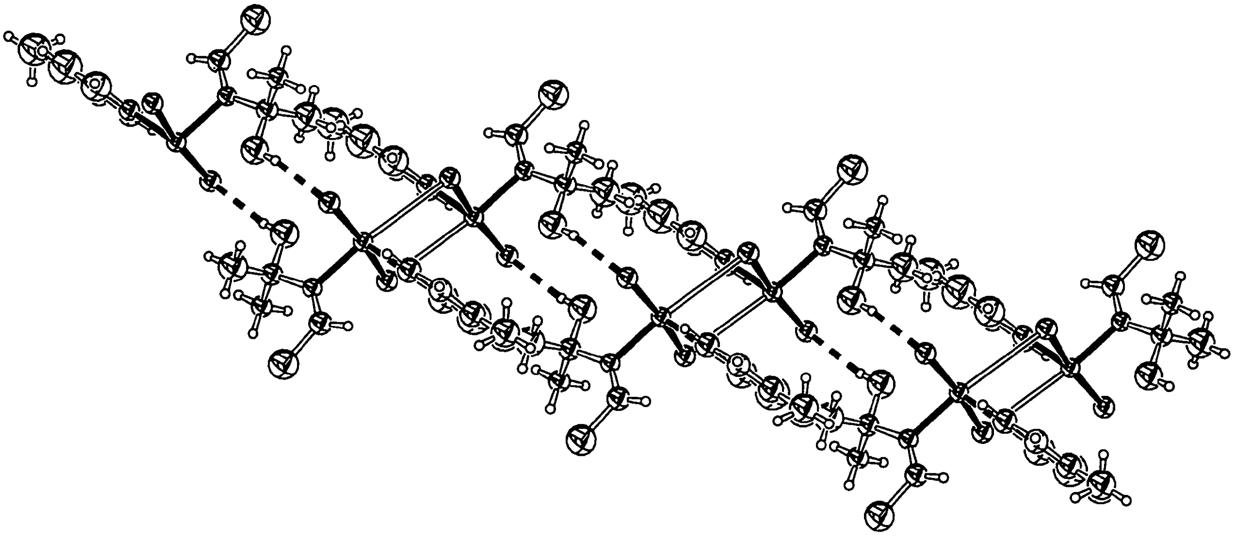


Fig. 2. The extended structure, with the hydrogen bonds represented by dashed lines and the long Te···Cl interactions by open bonds. The zigzag chain runs parallel to the *b* axis.

Experimental

2-Methyl-3-butyn-2-ol was reacted with *p*-methoxyphenyltellurium trichloride in benzene under reflux. Recrystallization of the crude mixture from dichloromethane allowed the separation of one isomer as colorless crystals suitable for X-ray diffraction; the other component remained in solution.

Crystal data

$C_{12}H_{15}Cl_3O_2Te$
$M_r = 425.19$
Triclinic
$P\bar{1}$
$a = 8.3743\,(4)$ Å
$b = 9.6681\,(7)$ Å
$c = 10.7682\,(9)$ Å
$\alpha = 100.195\,(6)°$
$\beta = 104.465\,(5)°$
$\gamma = 105.408\,(5)°$
$V = 785.71\,(9)$ Å^3
$Z = 2$
$D_x = 1.797$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.17$–$20.93°$
$\mu = 2.394$ mm^{-1}
$T = 293\,(2)$ K
Irregular
0.30 × 0.20 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.534$, $T_{\max} = 0.646$
3155 measured reflections
2939 independent reflections

2723 reflections with $F^2 > 2\sigma F^2$
$R_{\rm int} = 0.009$
$\theta_{\max} = 25.57°$
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 12$
3 standard reflections
frequency: 30 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.121$
2939 reflections
172 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.2325P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.807$ e Å^{-3}
$\Delta\rho_{\min} = -0.969$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Te—C1	2.124 (3)	Te—Cl1	2.4820 (8)
Te—C6	2.129 (3)	Te—Cl2	2.5600 (8)
C1—Te—C6	98.02 (12)	C1—Te—Cl2	87.06 (8)
C1—Te—Cl1	88.03 (8)	C6—Te—Cl2	92.42 (8)
C6—Te—Cl1	92.86 (8)	Cl1—Te—Cl2	173.24 (3)

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.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1059). Services for accessing these data are described at the back of the journal.

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Two isomers of 1,4a,5,7,8,9,9a,10,11,12a-decahydro-1,4-methanocyclopenta[*b*]pyrrolo[1,6a-*a*][3,1]benzoxazin-11-one

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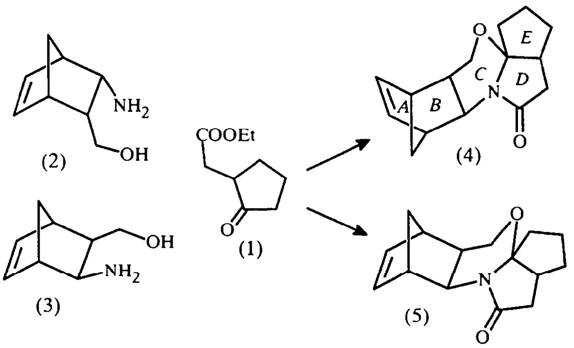
Abstract

The reactions of 2-ethoxycarbonylmethylcyclopentanone with *diendo*- or *diexo*-3-aminobicyclo[2.2.1]hept-5-ene-2-methanol yielded isomeric cyclopentan[*b*]pyrrolo[1,6a-*a*][3,1]benzoxazinones. The crystal structures of the two isomers ($C_{15}H_{19}NO_2$) were elucidated by means of X-ray crystallography.

Comment

2-Aroylcyclohexanecarboxylic acids were earlier used to prepare tetra- and pentacyclic compounds containing two hetero rings and one or two cycloalkane rings at the terminal(s), with isoindolone or indolone as the central structural unit (Stájer *et al.*, 1994, 1996; Sillanpää *et al.*, 1997; Virág *et al.* 1998). The aim of these syntheses was to obtain pharmacologically active compounds. The preparation of the saturated derivatives yielded isomeric compounds with different stereostructures. Similar reactions of β-oxocycloalkanecarboxylates afforded condensed-skeleton bicyclic lactams, thiophenes, pyrones *etc.* (Omar & Frahm, 1990; Noe *et al.*, 1986; Hua *et al.*, 1990; Kraatz, 1984).

The above reactions have now been extended to those between 2-ethoxycarbonylmethylcyclopentanone, (1), and *diendo*-, (2), and *diexo*-3-aminobicyclo[2.2.1]hept-5-ene-2-methanol, (3). Whereas the earlier applied γ-oxocarboxylic acids, such as 2-aroylcyclohexanecarboxylic acid, led to isoindolones, (1) yielded saturated norbornene and cyclopentane-condensed pyrrolones. The differences from the similar compounds where ring *A* was *cis*- or *trans*-fused cyclohexane or cyclohexene, and ring *E* either cyclopentane or cyclohexane (Virág *et al.*, 1998), are as follows: (*a*) ring *A* is methylene-bridged and fused *diendo* or *diexo* here, always containing a double bond, and (*b*) ring *E* is now a fused cyclopentane moiety.



Compound (1), prepared by a literature method (Mondon, 1959), was reacted with *diendo*-, (2), or *diexo*-3-aminobicyclo[2.2.1]hept-5-ene-2-methanol, (3) (Stájer *et al.*, 1983, 1984), to yield methanocyclopenta[*b*]pyrrolo[1,6a-*a*][3,1]benzoxazinones [(4) and (5)]. As (4) and (5) gave overlapping signals, the stereostructures could not be established by NMR methods. X-ray analysis, however, allowed elucidation of the structures. Perspective views of (4) and (5) are depicted in Figs. 1 and 2, respectively. The measurements

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