

**Data collection**

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.042$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.97^\circ$
Absorption correction: none	$h = -1 \rightarrow 11$
4100 measured reflections	$k = -1 \rightarrow 16$
3163 independent reflections	$l = -16 \rightarrow 16$
1754 reflections with $I > 2\sigma(I)$	3 standard reflections
	frequency: 60 min
	intensity decay: none

**Refinement**

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R(F) = 0.040$	$\Delta\rho_{\text{max}} = 0.480 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.086$	$\Delta\rho_{\text{min}} = -0.365 \text{ e } \text{\AA}^{-3}$
$S = 1.116$	Extinction correction: none
3163 reflections	Scattering factors from
199 parameters	International Tables for
H atoms constrained	Crystallography (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.5754P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Selected bond lengths (Å)

S1—C1	1.755 (4)	S6—C13	1.737 (5)
S1—C3	1.776 (5)	S6—C12	1.756 (5)
S2—C2	1.726 (5)	S7—C9	1.740 (5)
S2—C3	1.767 (4)	S7—C8	1.854 (4)
S3—C4	1.757 (4)	S8—C10	1.744 (5)
S3—C5	1.818 (5)	S8—C8	1.839 (4)
S4—C4	1.760 (4)	C1—C2	1.347 (6)
S4—C6	1.810 (5)	C1—C11	1.439 (6)
S5—C14	1.730 (5)	C3—C4	1.326 (6)
S5—C12	1.761 (4)		

Data collection: CAD-4-PC (Enraf–Nonius, 1994). Cell refinement: CAD-4-PC. Data reduction: XCAD4 (Harms, 1993). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: SHELXL93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1493). Services for accessing these data are described at the back of the journal.

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**Dibromo[(Z)-2-bromo-2-(hydroxymethyl)-vinyl](n-butyl)tellurium(IV)**

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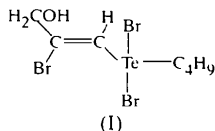
**Abstract**

In the title compound, [TeBr<sub>2</sub>(C<sub>4</sub>H<sub>9</sub>)(C<sub>3</sub>H<sub>4</sub>BrO)] or C<sub>7</sub>H<sub>13</sub>Br<sub>3</sub>OTe, the primary geometry about the Te<sup>IV</sup> atom is a pseudo-trigonal-bipyramidal arrangement with axial bromines and one equatorial position vacant. If an intermolecular secondary Te···O bond of 3.041 (5) Å, *trans* to the Te—C(vinyl) bond, is taken into consideration, the structure may be regarded as pseudo-octahedral. Distances and angles are: Te—Br 2.7295 (10) and 2.6115 (10), Te—C 2.092 (6) and 2.134 (7) Å, Br—Te—Br 176.59 (3), Br—Te—C 88.87 (19), 89.0 (3), 89.65 (19) and 88.2 (3), and C—Te—C 99.7 (3)°. The lattice is formed by hydrogen-bonded centrosymmetric dimers [O···Br = 3.399 (6) Å], which, in turn, are linked by the intermolecular secondary Te···O bond.

**Comment**

Vinyl tellurides are intermediates in the synthesis of vinyl lithium compounds, which are, in turn, important intermediates in organic synthesis, either as precursors of the widely used vinyl cuprate compounds or as nucleophiles leading to chain-elongation products by

reaction with many electrophiles (for a review see Comasseto *et al.*, 1997). The synthetic route led to the production of the *Z* and *E* isomers of the title compound, (I), and only one of them was crystallized. As the regio- and stereochemistry of the isomers could not be determined by the usual spectroscopic methods, a crystal structure determination was undertaken.



The crystal structure shows that the *Z* isomer was the one crystallized. The Te<sup>IV</sup> atom adopts a primary four-coordinate geometry based on a pseudo-trigonal-bipyramidal polyhedron formed by four bonds to two Br and two C atoms of the ligands and one lone pair of electrons. The lone pair and the C atoms occupy equatorial sites, while the Br atoms occupy axial positions. The Te—Br bond lengths are very close to the longest and shortest values already found in reported structures having a C<sub>2</sub>—Te—Br<sub>2</sub> subunit, and the Br—Te—Br angle is in the range observed in other compounds (Farran *et al.*, 1995). The Te1—C1 distance is in good agreement with the sum of the Pauling (1960) single-bond covalent radii of Te (1.37 Å) and C<sub>sp</sub><sup>2</sup> (0.74 Å) atoms, and with the values of 2.073 (7), 2.109 (5) and 2.127 (3) Å reported in Zukerman-Schpector *et al.* (1995), Zukerman-Schpector, Caracelli, Dabdoub & Dabdoub (1996), and Zukerman-Schpector, Caracelli, Dabdoub, Dabdoub & Pereira (1996), respectively. The Te1—C4 distance is in good agreement with the sum of the Pauling (1960) single-bond covalent radii for a Te—C<sub>sp</sub><sup>3</sup> single-covalent bond (2.142 Å).

The C1···C3 distance of 2.53 (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'Keefe & Hyde, 1981). From the Te···C2 distance of 3.026 (5) Å, the non-bonded radius for the Te atom may be estimated to be 1.78 Å, a value which is in good agreement, to within experimental accuracy, with those found in related compounds (Zukerman-Schpector, Caracelli, Dabdoub & Dabdoub, 1996, and references therein). On the other hand, the C6—C7 bond length is rather short. The rather large values of the *U*<sup>11</sup>, *U*<sup>22</sup> and *U*<sup>12</sup> displacement tensor elements of the C7 atom cause the distance between the mean atomic positions to be less than the corresponding mean interatomic separation, which is the quantity of chemical significance (Johnson, 1970).

The crystal lattice is built of centrosymmetric hydrogen-bonded dimers [O1···Br1<sup>ii</sup> = 3.399 (6), H1O1···Br1<sup>iii</sup> = 2.596 Å and O1—H1O1···Br1<sup>iii</sup> = 166.3°; symmetry code: (ii) 1 - *x*, 1 - *y*, 1 - *z*]. These, in turn, are linked through a secondary interaction Te1···O1<sup>i</sup> of 3.040 (5) Å [symmetry code: (i) -1 + *x*, *y*,

*z*], which is shorter than the sum of the van der Waals radii of Te and O (3.6 Å; Pauling, 1960). With this interaction, which is approximately *trans* to the vinylic C atom [O1<sup>i</sup>···Te1—C1 = 169.1 (2)°], the arrangement about the Te atom should be described as a distorted pseudo-octahedron, with the lone pair of electrons occupying the sixth position, as shown in Fig. 1.

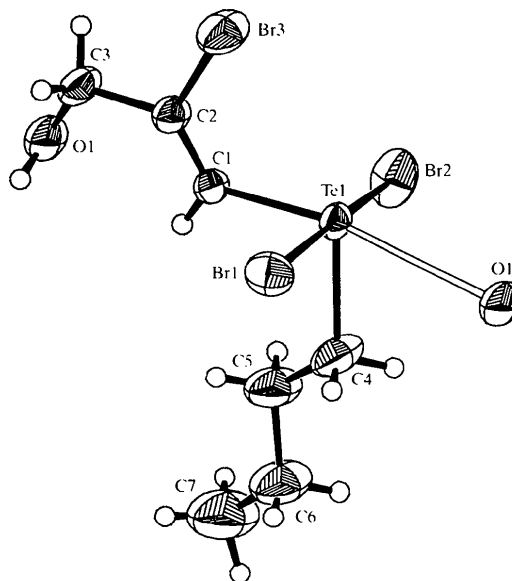


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. Atom O1<sup>i</sup> was generated by the symmetry operation *x* - 1, *y*, *z*.

## Experimental

Butyltellurium tribromide (2 mmol) was added to hydroxymethylacetylene (2 mmol) in benzene (20 ml) and the resulting solution refluxed for 6.5 h. Crystals were obtained by slow evaporation from hexane/ethyl acetate at 269 K.

### Crystal data

C<sub>7</sub>H<sub>13</sub>Br<sub>3</sub>OTe  
*M<sub>r</sub>* = 480.491  
 Triclinic  
*P*1̄  
*a* = 7.9543 (5) Å  
*b* = 8.9620 (10) Å  
*c* = 9.4344 (9) Å  
 $\alpha$  = 90.360 (9)°  
 $\beta$  = 105.946 (6)°  
 $\gamma$  = 95.091 (7)°  
*V* = 643.8 (1) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 2.4786 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Enraf-Nonius CAD-4  
 diffractometer

Mo K $\alpha$  radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25  
 reflections  
 $\theta$  = 9.67–18.11°  
 $\mu$  = 11.582 mm<sup>-1</sup>  
*T* = 293 K  
 Irregular  
 0.25 × 0.20 × 0.15 mm  
 Yellow

1765 reflections with  
 $F^2 > 2\sigma(F^2)$

$\omega/2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (North *et al.*,  
 1968)  
 $T_{\min} = 0.094$ ,  $T_{\max} = 0.176$   
 2552 measured reflections  
 2388 independent reflections

$R_{\text{int}} = 0.014$   
 $\theta_{\text{max}} = 25.46^\circ$   
 $h = -9 \rightarrow 9$   
 $k = 0 \rightarrow 10$   
 $l = -11 \rightarrow 11$   
 3 standard reflections  
 frequency: 30 min  
 intensity decay: 0.5%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.078$   
 $S = 1.085$   
 2388 reflections  
 112 parameters  
 H-atom parameters  
 constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 1.5688P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.776 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.764 \text{ e } \text{\AA}^{-3}$   
 Extinction correction:  
*SHELXL97* (Sheldrick,  
 1997)  
 Extinction coefficient:  
 0.0085 (7)  
 Scattering factors from  
*International Tables for*  
*Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Te1—C1	2.092 (6)	C1—C2	1.308 (9)
Te1—C4	2.134 (7)	C2—C3	1.523 (9)
Te1—Br2	2.6115 (10)	C4—C5	1.445 (10)
Te1—Br1	2.7295 (10)	C5—C6	1.502 (11)
Br3—C2	1.889 (6)	C6—C7	1.425 (13)
O1—C3	1.403 (8)		
C1—Te1—C4	99.7 (3)	Br2—Te1—Br1	176.59 (3)
C1—Te1—Br2	88.87 (19)	C2—C1—Te1	124.1 (5)
C4—Te1—Br2	89.0 (3)	Br1—Te1—O1 <sup>1</sup>	82.5 (1)
C1—Te1—Br1	89.65 (19)	Br2—Te1—O1 <sup>1</sup>	98.5 (1)
C4—Te1—Br1	88.2 (3)	C4—Te1—O1 <sup>1</sup>	72.6 (2)

Symmetry code: (i)  $x - 1, y, z$ .

H atoms were refined with fixed geometry, each riding on a carrier atom, with an isotropic displacement parameter amounting to 1.5 (for methyl H atoms) or 1.2 (for the other H atoms) times the value of the equivalent isotropic displacement parameter of the atom to which they are attached. The hydroxyl H atom was found in a difference Fourier map and the torsion around the O atom refined.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1481). Services for accessing these data are described at the back of the journal.

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## 7-Methoxy-1-[1-(6-methoxy-2-naphthoyl)-ethyl]-2-methyl-2,3-dihydro-1H-cyclopenta[*a*]naphthalen-3-one

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### Abstract

The structure of the title compound,  $\text{C}_{29}\text{H}_{26}\text{O}_4$ , prepared from 1-(6-methoxy-2-naphthyl)propan-1-one and ethyl *ortho*-formate in the presence of boron trifluoride under microwave irradiation, consists of connected 7-methoxy-2-methyl-2,3-dihydro-1H-cyclopenta[*a*]naphthalen-3-one and 1-(6-methoxy-2-naphthyl)propan-1-one units. The space group, *Pc*, is non-centrosymmetric but achiral; three chiral centres exist in the molecule, but both enantiomers are present in the crystal.