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# Structure of Dibromo(2-methoxycyclohexyl)phenyltellurium

BY RAJ K. CHADHA* AND THI NGUYEN

*Department of Chemistry, University of Manitoba, Winnipeg, MB, Canada R3T 2N2*

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**Abstract.** $[TeBr_2(C_6H_5)(C_7H_{13}O)]$, $M_r = 477{\cdot}4$, orthorhombic, *Pbca*, $a = 32{\cdot}683\,(9)$, $b = 10{\cdot}962\,(3)$, $c = 8{\cdot}830\,(3)$ Å, $V = 3164\,(2)$ Å$^3$, $Z = 8$, $D_x = 2{\cdot}00$, $D_m = 2{\cdot}02$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 66{\cdot}85$ cm$^{-1}$, $F(000) = 1808$, $T = 294\,(1)$ K, $R = 0{\cdot}0298$ for 1470 reflections $[I > 3\sigma(I)]$. The phenyl and cyclohexyl rings are present at the equatorial and the Br atoms at the axial positions in a pseudo trigonal bipyramidal geometry around the Te atom. The mean Te—Br and Te—C distances are 2·673 (3) and 2·16 (2) Å respectively. Tellurium is involved in one intermolecular (Te···Br) and one intramolecular (Te···O) secondary-bond interaction. $PhTeBr_2$ and $OCH_3$ groups occupy equatorial positions on adjacent C atoms of the cyclohexyl ring, so that the compound has *trans* geometry.

**Introduction.** $ArTeX_3$ ($X$ = Cl, Br) reacts with terminal and cyclic olefins in $CH_3OH$ to give ($\beta$-methoxyalkyl)aryltellurium dihalides (Uemura & Fukuzawa, 1983). The reaction is regiospecific in the case of terminal olefins, and the addition of $ArTeX_2$ and $OCH_3$ groups to the olefins occurs in a *trans* fashion (Uemura, Fukuzawa & Toshimitsu, 1983). To understand the conformation of the product obtained from the addition of an $ArTeX_3$ to a cyclic olefin, a crystallographic analysis of dibromo-(2-methoxycyclohexyl)phenyltellurium, (1), was undertaken, the results of which are reported here.

**Experimental.** Preparation as described by Uemura, Fukuzawa & Toshimitsu (1983), followed by slow evaporation of $CH_2Cl_2$, density measured by flotation in $CCl_4/CH_3I$. A single crystal was mounted along the largest dimension, subsequently shown to be the $c$ axis. Data were collected on a Syntex $P2_1$ diffractometer. Lattice parameters from 15 strong reflections, $15 < 2\theta < 30°$. The space group *Pbca* was determined from Laue symmetry and systematic absences, $0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h = 2n + 1$. Three monitor reflections measured after every 100 reflections indicated no decay over 66 h of X-ray exposure. Lorentz and polarization corrections applied; analytical absorption correction with *ABSORB* (Templeton & Templeton, 1973). Min. and max. corrections and other relevant details are given in Table 1. Position of the Te atom was obtained from a sharpened Patterson synthesis. Remaining non-H atoms revealed from $\Delta F$ calculations. Structure refined anisotropically by full-matrix least-squares methods. H atoms included in idealized positions (C—H 0·95 Å and CCH 120 or 109·5°). Refinement minimizing the function $\sum w(|F_o| - |F_c|)^2$ converged to $R = 0{\cdot}0298$ and $wR = 0{\cdot}0320$. Weights were assigned as $w = 1/[\sigma^2(F) + pF^2]$, with a final $p$ value of 0·00001. Computation was carried out on the University of Manitoba Computer Services Department's Amdahl 580/5850 mainframe computer. Scattering factors for all non-H atoms, including anomalous-dispersion correction for Te and Br, obtained from Ibers & Hamilton (1974), for H atoms from Stewart, Davidson & Simpson (1965). Programs used include *SHELX76* (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *ORTEP* (Johnson, 1965). The atomic coordinates and equivalent parameters for non-H atoms are given in Table 2 and distances and angles in Table 3.†

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* Author to whom all the correspondence should be addressed. Present address: Department of Chemistry, B-003, University of California, San Diego, La Jolla, CA 92093, USA.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52198 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Summary of intensity collection and structural refinement*

| | |
|---|---|
| Crystal dimensions (mm), color | 0·17 × 0·35 × 0·57, colorless |
| Min. absorption correction | 2·875 |
| Max. absorption correction | 9·227 |
| Monochromator | Highly oriented graphite |
| $2\theta$ range (°) | 4–45 |
| Scan type | $\theta/2\theta$ |
| Scan width (°) | $2\theta_{\alpha1} - 1$ to $2\theta_{\alpha2} + 1$ |
| Scan speed ($2\theta$° min$^{-1}$) | Variable, 2·02–4·88 |
| Background time/scan time | 0·5 |
| Total reflections measured | 2510 ($h$ 0→34, $k$ 0→11, $l$ 0→9) |
| Unique data used | 1470 [$I > 3\sigma(I)$] |
| No. of parameters | 154 |
| $R$ | 0·0298 |
| $wR$ | 0·0320 |
| $\Delta\rho$ (e Å$^{-3}$) | 0·4, −0·3 |
| Max. shift/e.s.d. | 0·01 |

Table 2. *Final fractional coordinates and equivalent isotropic thermal parameters* (×10³) *with e.s.d.'s in parentheses for non*-H *atoms*

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

| | $x$ | $y$ | $z$ | $U_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Te | 0·11792 (2) | 0·61303 (4) | 0·20272 (7) | 38·1 (4) |
| Br(1) | 0·0645 (1) | 0·4459 (1) | 0·3013 (1) | 58 (4) |
| Br(2) | 0·1658 (1) | 0·7874 (1) | 0·0881 (1) | 59 (1) |
| O | 0·0604 (2) | 0·6967 (5) | −0·0415 (8) | 49 (5) |
| C(1) | 0·1099 (3) | 0·5427 (7) | −0·0267 (10) | 39 (5) |
| C(2) | 0·0671 (3) | 0·5714 (7) | −0·0743 (10) | 42 (6) |
| C(3) | 0·0601 (3) | 0·5375 (8) | −0·2395 (12) | 57 (7) |
| C(4) | 0·0720 (4) | 0·4040 (8) | −0·2672 (12) | 80 (8) |
| C(5) | 0·1152 (4) | 0·3772 (9) | −0·2155 (11) | 72 (7) |
| C(6) | 0·1207 (3) | 0·4087 (7) | −0·0511 (11) | 52 (6) |
| C(7) | 0·0195 (3) | 0·7345 (9) | −0·0429 (15) | 79 (7) |
| C(11) | 0·1695 (3) | 0·5014 (7) | 0·2613 (10) | 41 (5) |
| C(12) | 0·1684 (3) | 0·4280 (8) | 0·3844 (12) | 55 (6) |
| C(13) | 0·2007 (4) | 0·3558 (9) | 0·4226 (14) | 75 (8) |
| C(14) | 0·2353 (4) | 0·3583 (11) | 0·3332 (16) | 82 (9) |
| C(15) | 0·2361 (4) | 0·4317 (11) | 0·2109 (17) | 91 (9) |
| C(16) | 0·2039 (3) | 0·5039 (9) | 0·1694 (12) | 65 (7) |

**Discussion.** The crystals of (1) consist of discrete molecules as shown in Fig. 1. The compound adopts a primary four-coordinate geometry about tellurium that may be considered as based on a distorted trigonal bipyramid with an equatorial lone pair. The Br atoms occupy axial and C atoms equatorial positions. Te—C and Te—Br bond lengths and C—Te—C and C—Te—Br bond angles (Table 3) are within the ranges found for other $R_2TeBr_2$ compounds (Chadha & Drake, 1984). The Te atom is involved in one intramolecular interaction with an O atom of the methoxy group [Te···O distance 3·004 (5) Å] and one weak intermolecular interaction with a Br atom of the neighboring molecule [Te···Br(2′) distance 3·901 (2) Å]. These interactions complete a distorted octahedral configuration about tellurium, as has been found in many structures of $R_2TeX_2$ ($X$ = Cl, Br, I), and link the molecules into infinite chains parallel to the crystallographic $c$ axis, which results in crystal growth along this axis. A view of the structure normal to the chains is shown

Table 3. *Interatomic distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Te—Br(1) | 2·676 (1) | Br(1)—Te—Br(2) | 174·8 (1) |
| Te—Br(2) | 2·670 (1) | Br(1)—Te—Cl(1) | 88·9 (2) |
| Te—C(1) | 2·183 (9) | Br(2)—Te—C(1) | 88·4 (2) |
| Te—C(11) | 2·146 (9) | Br(1)—Te—C(11) | 92·5 (3) |
| O—C(2) | 1·420 (9) | Br(2)—Te—C(11) | 92·3 (3) |
| O—C(7) | 1·40 (1) | C(1)—Te—C(11) | 96·7 (3) |
| C(1)—C(2) | 1·49 (1) | C(2)—O—C(7) | 115·5 (7) |
| C(1)—C(6) | 1·53 (1) | Te—C(1)—C(2) | 107·4 (6) |
| C(2)—C(3) | 1·52 (1) | Te—C(1)—C(6) | 116·3 (6) |
| C(3)—C(4) | 1·53 (1) | C(2)—C(1)—C(6) | 112·3 (7) |
| C(4)—C(5) | 1·51 (2) | O—C(2)—C(1) | 106·9 (7) |
| C(5)—C(6) | 1·50 (1) | O—C(2)—C(3) | 114·1 (7) |
| C(11)—C(12) | 1·35 (1) | C(1)—C(2)—C(3) | 110·0 (8) |
| C(11)—C(16) | 1·39 (1) | C(2)—C(3)—C(4) | 110·4 (8) |
| C(12)—C(13) | 1·36 (1) | C(3)—C(4)—C(5) | 111·9 (8) |
| C(13)—C(14) | 1·38 (2) | C(4)—C(5)—C(6) | 111·0 (9) |
| C(14)—C(15) | 1·35 (2) | C(1)—C(6)—C(5) | 109·3 (8) |
| C(15)—C(16) | 1·37 (2) | Te—C(11)—C(12) | 120·8 (8) |
| Te···Br(2′) | 3·901 (2) | Te—C(11)—C(12) | 120·8 (8) |
| Te···O | 3·004 (5) | Te—C(11)—C(16) | 119·0 (7) |
| Br(1)—Te···Br(2′) | 99·8 (4) | C(12)—C(11)—C(16) | 120·2 (9) |
| Br(2)—Te···Br(2′) | 84·0 (4) | C(11)—C(12)—C(13) | 122 (1) |
| C(1)—Te···Br(2′) | 163·0 (8) | C(12)—C(13)—C(14) | 119 (1) |
| C(11)—Te···Br(2′) | 68·5 (8) | C(13)—C(14)—C(15) | 119 (1) |
| Br(1)—Te···O | 92·0 (6) | C(14)—C(15)—C(16) | 123 (1) |
| Br(2)—Te···O | 82·9 (6) | C(11)—C(16)—C(15) | 117 (1) |
| C(1)—Te···O | 50·7 (8) | | |
| C(11)—Te···O | 147·0 (8) | | |
| Br(2′)···Te···O | 142·3 (7) | | |

Symmetry-equivalent position: (′) $x$, $1·5 - y$, $0·5 + z$.

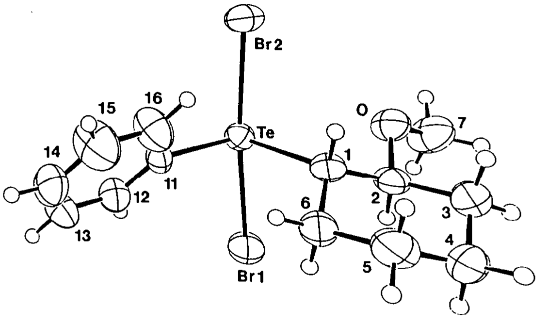

Fig. 1. *ORTEP* plot of (1). The non-H atoms are contoured at 50% probability; H atoms are drawn arbitrarily small. The labeling scheme is defined; atoms with numbers only are C atoms.

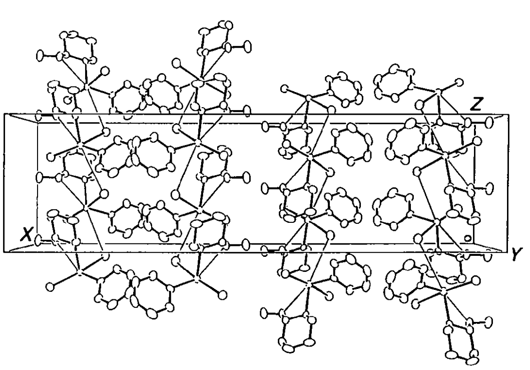

Fig. 2. View normal to the chains formed by the Te···Br secondary bonds, which are shown by thin bonds. The ellipsoids correspond to 20% probability. The H atoms are omitted for clarity.

in Fig. 2. The intermolecular bonding arrangement in the present structure is thus similar to that in bis(*p*-trimethylsilyltolyl)tellurium dichloride (Chadha & Drake, 1984), phenoxatellurin 10,10-diiodide (McCullough, 1973), dibenzotellurophene diiodide (McCullough, 1975) and $\alpha$-1,1-diiodo-3,4-benzo-1-telluracyclopentane (Knobler & Ziolo, 1979) in which the chains are parallel to the *c*, *a*, *b*, *c* axes respectively.

The cyclohexane ring adopts a regular chair conformation and the two substituents $OCH_3$ and $PhTeBr_2$, which are present at the 1,2 positions, occupy equatorial sites. This structure thus confirms the *trans* geometry of the compound as expected on the basis of bulkiness of the substituents. The average C—C distance of 1·51 Å, C—C—C bond angle of 110·8 (8)° and CCCC torsion angle of 56·1 (6)° are quite close to the cyclohexane values of 1·53 (1) Å, 111·6 (2) and 54·5 (8)° obtained from electron diffraction (Davis & Hassel, 1963).

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# Formation of Diastereoisomeric Pinacols *via* Reductive Coupling of $D_3$-Trishomocubanone

By William H. Watson* and Ante Nagl†

*Materials Research Laboratory, Department of Chemistry, Texas Christian University, Fort Worth, Texas* 76129, *USA*

and Alan P. Marchand* and G. Madhusudhan Reddy

*Department of Chemistry, University of North Texas, Box* 5068, *Denton, Texas* 76203, *USA*

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**Abstract.** Spiro[pentacyclo[7.3.0.$0^{4,8}$.$0^{5,12}$.$0^{7,11}$]dodecan-2-one-3,11′-pentacyclo[6.3.0.$0^{2,6}$.$0^{3,10}$.$0^{5,9}$]undecane] (4*b*), $C_{22}H_{24}O$, $M_r = 303·43$, monoclinic, $C2/c$, $a = 20·593$ (3), $b = 6·645$ (1), $c = 26·061$ (4) Å, $\beta = 121·78$ (1)°, $V = 3032·3$ (9) Å$^3$, $Z = 8$, $D_x = 1·133$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·74$ cm$^{-1}$, $F(000) = 1312$, $T = 298$ K, $R = 0·0439$ for 1731 reflections. Compound (4*b*) consists of two spiro-fused cage systems. One cage is composed of three fused norbornane (bicyclo[2.2.1]heptane) moieties, while the other contains two norbornane and one bicyclo[2.2.2]octanone moiety. The spiro fusion involves the methano bridge of one norbornane and the ethano bridge of the bicyclo[2.2.2]octane group. The ideal envelope conformations of the five-membered rings comprising the norbornane moieties are significantly twisted by the ring fusions with the normal 0° torsion angles from 16·6 (2) to 28·5 (2)°. The bicyclooctane system also exhibits large twist distortions.

**Introduction.** As part of a program that is concerned with the synthesis and chemistry of novel polycyclic

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* Authors to whom correspondence should be addressed.

† On leave from the Faculty of Technology, University of Zagreb, Zagreb, Yugoslavia.

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