

Table 2. Selected interatomic distances (Å) and bond angles (°)

Sn(1)—O(1)	2.021 (3)	Sn(1)—O(2)	2.149 (3)
Sn(1)—Cl	2.485 (1)	Sn(1)—C(111)	2.132 (2)
Sn(1)—C(121)	2.129 (6)	Sn(2)—O(1)	2.048 (3)
Sn(2)—O(2)	2.138 (3)	Sn(2)—O(1')	2.121 (3)
Sn(2)—C(211)	2.127 (5)	Sn(2)—C(221)	2.110 (5)
O(2)···O(1)	2.729 (7)	H(2)···O(1)	1.80 (1)
O(1)—Sn(1)—O(2)	72.8 (1)	O(1)—Sn(1)—Cl	86.2 (1)
O(1)—Sn(1)—C(111)	115.3 (1)	O(1)—Sn(1)—C(121)	112.9 (2)
O(2)—Sn(1)—Cl	159.0 (1)	O(2)—Sn(1)—C(111)	93.9 (2)
O(2)—Sn(1)—C(121)	94.2 (2)	Cl—Sn(1)—C(111)	95.1 (2)
Cl—Sn(1)—C(121)	94.0 (2)	C(111)—Sn(1)—C(121)	131.4 (2)
O(1)—Sn(2)—O(2)	72.5 (1)	O(1)—Sn(2)—O(1')	73.4 (3)
O(1)—Sn(2)—C(211)	116.5 (2)	O(1)—Sn(2)—C(221)	117.5 (2)
O(1')—Sn(2)—O(2)	146.0 (1)	O(1')—Sn(2)—C(211)	99.9 (3)
O(1')—Sn(2)—C(221)	99.5 (3)	O(2)—Sn(2)—C(211)	95.8 (2)
O(2)—Sn(2)—C(221)	95.4 (2)	C(211)—Sn(2)—C(221)	125.8 (2)
Sn(1)—O(1)—Sn(2)	111.4 (1)	Sn(1)—O(1)—Sn(2')	142.1 (1)
Sn(2)—O(1)—Sn(2')	106.6 (1)	Sn(1)—O(2)—Sn(2)	103.3 (1)
O(2)—H(2)···O(1)	159 (1)		

Primed atoms are related by centre of inversion.

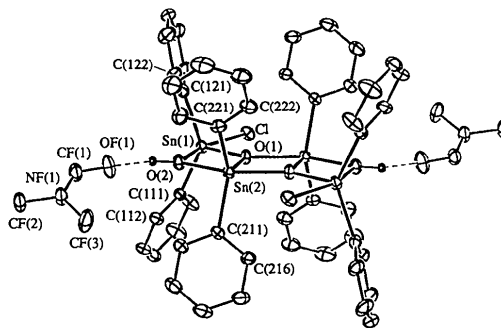


Fig. 1. Molecular structure and crystallographic numbering scheme employed for [Sn<sub>2</sub>(Cl)(O)(OH)(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>]<sub>2</sub>·2C<sub>3</sub>H<sub>7</sub>NO (Johnson, 1971).

0.022,  $wR = 0.025$ ,  $w = [\sigma^2(F) + 0.001|F|^2]^{-1}$ ,  $S = 1.16$ ,  $(\Delta/\sigma)_{\max} \leq 0.002$ ,  $\Delta\rho_{\max} = 0.36$ ,  $\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$ ; no extinction correction applied. Scattering factors for all atoms were as incorporated in *SHELX76* (Sheldrick, 1976). All calculations were performed on a Sun4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2\* and the numbering scheme used is shown in Fig. 1, which was drawn with *ORTEP* (Johnson, 1971) at 15% probability levels.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all interatomic parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53533 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of 1,4,5,8,9,10-Hexabromo-11,11-dimethoxytricyclo[6.2.1.0<sup>2,7</sup>]undeca-4,9-diene-3,6-dione

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**Abstract.** C<sub>13</sub>H<sub>8</sub>Br<sub>6</sub>O<sub>4</sub>,  $M_r = 707.7$ , orthorhombic, *Pna*2<sub>1</sub>,  $a = 12.711 (5)$ ,  $b = 16.921 (4)$ ,  $c = 8.455 (3) \text{ \AA}$ ,  $V = 1818.5 (11) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 2.584 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 131.4 \text{ cm}^{-1}$ ,  $F(000) = 1312$ ,  $T = 100 (4) \text{ K}$ , final  $R =$

0108-2701/91/030662-03\$03.00

**Related literature.** Closely related structures have been reported for [(*R*<sub>4</sub>Sn<sub>2</sub>ClO<sub>2</sub>H)<sub>2</sub>], for  $R = i\text{-C}_3\text{H}_7$  (Puff, Bung, Friedrichs & Jansen, 1983) and  $R = \text{C}_6\text{H}_5$ , as an acetone solvate (Vollano *et al.*, 1984).

The Australian Research Council is thanked for support.

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0.043 for 883 reflections with  $I > 3\sigma(I)$ . The title compound is an *endo* addition product of 2,3-dibromobenzoquinone and 1,2,3,4-tetrabromo-5,5-dimethoxycyclopentadiene. The two remaining double bonds are parallel and separated by

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3.34 (3) Å, a configuration which allows further intramolecular cyclization. The shortest intermolecular contact is 2.91 (1) Å between Br(5) and O(2).

**Experimental.** The title compound was synthesized *via* a [4 + 2] cycloaddition of 2,3-dibromobenzoquinone and 1,2,3,4-tetrabromo-5,5-dimethoxycyclopentadiene. A colorless needle-shaped crystal with approximate dimensions 0.6 × 0.3 × 0.3 mm was glued to a glass fiber and mounted on an Enraf-Nonius CAD-4 diffractometer with graphite crystal monochromator. Unit cell parameters were obtained from least-squares refinement of the setting angles of 25 reflections with  $24 < 2\theta < 42^\circ$  using Mo  $K\alpha$  radiation. Systematic absences,  $0kl: k + l = 2n + 1$  and  $h0l: h = 2n + 1$  indicated space group  $Pna2_1$  or  $Pnma$ . The non-centrosymmetric space group,  $Pna2_1$ , was chosen on the basis of intensity statistics and confirmed by successful determination of the structure. Intensities measured using  $\omega:2\theta$  scans within the ranges  $2 \leq 2\theta \leq 44^\circ$ ,  $0 \leq h \leq 13$ ,  $0 \leq k \leq 17$ ,  $0 \leq l \leq 8$ . The intensities of three standard reflections measured at 2 h intervals showed a variation of -5.3% during data collection and linear decay correction was applied. Absorption as a function of  $\psi$  was measured and an empirical absorption correction applied, with transmission coefficients ranging from 0.011 to 0.009. Lorentz and polarization corrections were applied to a total of 1196 measured reflections of which 883 were considered observed [ $I > 3\sigma(I)$ ].

The structure was determined by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980; modified by Frenz, 1982). Peaks corresponding to the six Br atoms were located in the  $E$  map and the positions of the remaining 17 non-H atoms were determined in a subsequent difference Fourier synthesis. The eight H-atom positions were calculated and added as a fixed contribution. Refinement was by full-matrix least-squares minimization of  $\sum w(|F_o| - |F_c|)^2$ , where  $w = 1/\sigma^2(F)$  and  $\sigma^2(F^2) = \sigma_{cs}^2 + (0.04F^2)^2$ . Positional and anisotropic thermal parameters were refined for the Br atoms, positional and isotropic thermal parameters were refined for all other atoms. The refinement converged at  $R = 0.045$ ,  $wR = 0.056$ ,  $S = 1.83$  and  $(\Delta/\sigma)_{\max} = 0.001$  for 114 variables. To test for the correct absolute configuration, the inversion operation was applied to the atomic coordinates and the refinement repeated. Final  $R = 0.043$ ,  $wR = 0.054$ ,  $S = 1.76$  and  $(\Delta/\sigma)_{\max} = 0.001$  indicating that the initial assignment of an absolute configuration was incorrect. Max. and min. peak heights in the final difference Fourier synthesis were 1.52 and  $-0.67 e \text{ \AA}^{-3}$ , respectively. Atomic scattering factors and anomalous-dispersion corrections taken from *International Tables for X-ray Crystallography* (1974,

Table 1. *Positional parameters and their estimated standard deviations*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
Br1	-0.0289 (2)	0.8333 (2)	0.240	3.18 (6)
Br2	0.1171 (2)	0.7537 (2)	0.5299 (4)	3.28 (5)
Br3	0.4242 (2)	0.9957 (2)	0.6425 (4)	3.23 (6)
Br4	0.3935 (2)	0.8603 (2)	0.3379 (4)	2.77 (5)
Br5	0.2427 (2)	0.9501 (2)	0.0283 (4)	2.75 (5)
Br6	0.1622 (2)	1.1364 (2)	0.1488 (4)	3.05 (6)
O1	-0.012 (1)	1.008 (1)	0.254 (2)	2.8 (3)*
O2	0.217 (1)	0.883 (1)	0.714 (2)	2.5 (4)*
O3	0.383 (1)	1.1184 (9)	0.320 (2)	2.5 (3)*
O4	0.273 (1)	1.1416 (9)	0.527 (2)	2.0 (3)*
C1	0.137 (2)	1.034 (1)	0.424 (3)	1.7 (5)*
C2	0.053 (2)	0.980 (1)	0.340 (3)	1.7 (5)*
C3	0.063 (2)	0.893 (1)	0.371 (3)	2.7 (6)*
C4	0.114 (2)	0.860 (1)	0.487 (3)	2.1 (5)*
C5	0.181 (1)	0.908 (1)	0.591 (2)	0.5 (4)*
C6	0.201 (2)	0.995 (1)	0.560 (3)	1.7 (5)*
C7	0.313 (2)	1.008 (1)	0.484 (3)	2.2 (5)*
C8	0.327 (2)	0.955 (2)	0.339 (3)	2.5 (5)*
C9	0.269 (2)	0.989 (1)	0.229 (3)	1.3 (4)*
C10	0.215 (2)	1.061 (1)	0.293 (3)	1.7 (5)*
C11	0.302 (2)	1.090 (1)	0.413 (3)	1.3 (4)*
C12	0.475 (2)	1.150 (2)	0.394 (3)	3.3 (6)*
C13	0.254 (2)	1.225 (2)	0.490 (3)	3.8 (7)*

Table 2. *Bond distances (Å) and bond angles (°)*

Numbers in parentheses are estimated standard deviations in the least significant digits.

Br1	C3	1.90 (2)	C7	C8	1.53 (3)		
Br2	C4	1.84 (2)	C7	C11	1.52 (3)		
Br3	C7	1.96 (2)	C8	C9	1.32 (3)		
Br4	C8	1.81 (2)	C1	C2	1.57 (3)		
Br5	C9	1.85 (2)	C1	C6	1.55 (3)		
Br6	C10	1.89 (2)	C1	C10	1.56 (3)		
O1	C2	1.20 (2)	C9	C10	1.50 (3)		
O2	C5	1.21 (2)	C2	C3	1.50 (3)		
O3	C11	1.39 (2)	C10	C11	1.57 (2)		
O3	C12	1.43 (2)	C3	C4	1.30 (3)		
C4	C5	1.46 (2)	O4	C11	1.35 (2)		
C5	C6	1.52 (3)	O4	C13	1.46 (2)		
C6	C7	1.58 (3)					
C11	O3	C12	119. (2)	Br3	C7	C11	116. (1)
C11	O4	C13	121. (2)	C6	C7	C8	110. (2)
C2	C1	C6	116. (2)	C6	C7	C11	102. (2)
C2	C1	C10	106. (2)	C8	C7	C11	103. (2)
C6	C1	C10	109. (1)	Br4	C8	C7	125. (2)
O1	C2	C1	121. (2)	Br4	C8	C9	130. (2)
O1	C2	C3	123. (2)	C7	C8	C9	104. (2)
C1	C2	C3	116. (2)	Br5	C9	C8	127. (2)
Br1	C3	C2	112. (1)	Br5	C9	C10	123. (1)
Br1	C3	C4	121. (2)	C8	C9	C10	110. (2)
C2	C3	C4	126. (2)	Br6	C10	C1	116. (1)
Br2	C4	C3	125. (2)	Br6	C10	C9	119. (1)
Br2	C4	C5	114. (1)	Br6	C10	C11	117. (1)
C3	C4	C5	121. (2)	C1	C10	C9	108. (2)
O2	C5	C4	123. (2)	C1	C10	C11	94. (1)
O2	C5	C6	115. (2)	C9	C10	C11	100. (1)
C4	C5	C6	122. (2)	O3	C11	O4	112. (2)
C1	C6	C5	117. (2)	O3	C11	C7	118. (2)
C1	C6	C7	96. (1)	O3	C11	C10	105. (1)
C5	C6	C7	111. (2)	O4	C11	C7	109. (2)
Br3	C7	C6	111. (1)	O4	C11	C10	119. (1)
Br3	C7	C8	114. (1)	C7	C11	C10	92. (1)

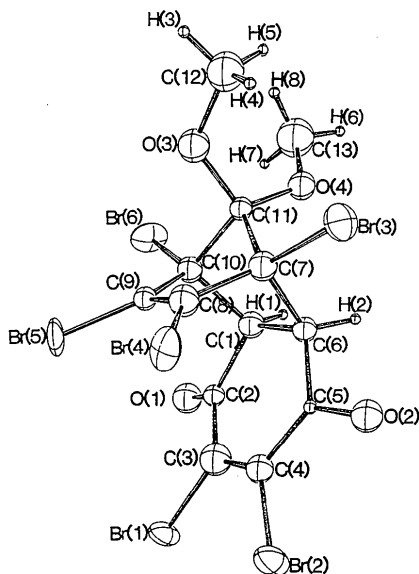


Fig. 1. ORTEP plot (Johnson, 1976) of the molecule showing the atomic numbering scheme. Thermal ellipsoids are plotted at the 50% probability level. H atoms are plotted with arbitrary radii.

Vol. IV), and all computer programs from the CAD4-SDP system (Frenz, 1982).<sup>\*</sup> Positional parameters and thermal parameters are given in Table 1, distances and angles in Table 2. Fig. 1 shows the atom numbering and Fig. 2 the unit-cell contents.

**Related literature.** The structure of the title compound is very similar to that of 2,5-dibromo-

<sup>\*</sup> Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53474 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

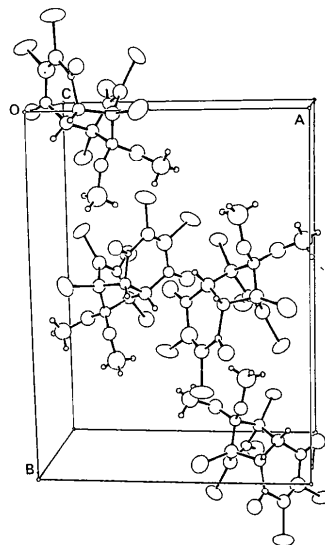


Fig. 2. Contents of the unit cell.

tricyclo[6.2.1.0<sup>2,7</sup>]undeca-4,9-diene-3,6-dione (Syed, Umrigar, Griffin & Stevens, 1984). Both compounds will undergo photo-induced [2 + 2] intramolecular cyclization to the corresponding trishomocubanes.

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## Structure of 7-Methoxy-5H-dibenz[c,e]azepine

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**Abstract.** C<sub>15</sub>H<sub>13</sub>NO, *M<sub>r</sub>* = 223.27, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 8.172 (6), *b* = 11.105 (8), *c* = 12.943 (7) Å, β = 99.83 (5)°, *V* = 1157 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.281 Mg m<sup>-3</sup>, *F*(000) = 472, λ(Mo Kα) =

0.71073 Å, μ = 0.08 mm<sup>-1</sup>, *T* = 291 (1) K, final *R* = 0.048 for 1311 unique observed [*F* ≥ 3.0σ(*F*)] diffractometer data. The central seven-membered ring has a boat conformation. The angle [41.47 (9)°] between

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