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2,2′-Dihydroxy-7,7,7′,7′-tetramethyl-*exo*-2,2′-bi{3,4-benzo-9-oxabicyclo[3.3.1]non-3-enyl}

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Abstract. $C_{28}H_{34}O_4$, orthorhombic, *Fdd*2, $a = 22{\cdot}771\ (14)$, $b = 19{\cdot}135\ (11)$, $c = 10{\cdot}308\ (5)$ Å, 23 °C; $M_r = 434{\cdot}58$, $Z = 8$, $D_x = 1{\cdot}285$ g cm^{-3}; the material was crystallized from acetone at room temperature. The molecule is located about a twofold axis; the two equivalent halves of the molecule are joined by a single C—C bond and by intramolecular hydrogen bonds.

Introduction. As an adjunct to continuing research on the photochemistry of cyclic dienones (see, for example, Hart & Suzuki, 1975*a*,*b*,*c*), irradiation of 7,7-dimethyl-3,4-benzo-9-oxabicyclo[3.3.1]non-3-en-2-one in cyclohexane through Pyrex gave a dimeric product for which attempts to elucidate its structure by use of IR, UV, and NMR spectra were not successful (Suzuki, 1975). The crystal and molecular structure determination of the title compound was undertaken to identify it.

Table 1. *Atomic parameters*

Fractional coordinates are $\times 10^4$, except those for hydrogen atoms, which are $\times 10^3$.

	x	y	z	B_{iso} (Å^2)
C(1)	5606 (1)	9659 (2)	6339 (5)	
C(2)	5226 (1)	9681 (2)	5104*	
C(3)	5646 (1)	9735 (2)	3962 (5)	
C(4)	6206 (1)	10012 (2)	4153 (5)	
C(5)	6416 (1)	10247 (2)	5468 (5)	
C(6)	6886 (2)	9782 (2)	6081 (6)	
C(7)	6624 (1)	9194 (2)	6920 (5)	
C(8)	6007 (1)	9020 (2)	6403 (5)	
O(9)	5932 (1)	10299 (1)	6360 (4)	
C(10)	5493 (2)	9479 (2)	2741 (5)	
C(11)	5885 (2)	9517 (2)	1721 (5)	
C(12)	6432 (2)	9805 (2)	1901 (5)	
C(13)	6589 (2)	10042 (2)	3097 (5)	
C(14)	6587 (2)	9409 (3)	8345 (5)	
C(15)	7014 (2)	8549 (3)	6834 (6)	
O(16)	4923 (1)	9035 (1)	4973 (4)	
H(1)	535 (1)	971 (1)	709 (3)	0·9 (5)
H(2)	656 (1)	1071 (2)	540 (3)	2·0 (6)
H(3)	717 (2)	1007 (2)	660 (4)	3·8 (8)
H(4)	713 (2)	957 (2)	536 (4)	3·9 (8)
H(5)	582 (1)	869 (2)	699 (3)	2·2 (6)
H(6)	606 (1)	882 (2)	548 (4)	3·4 (8)
H(7)	509 (2)	926 (2)	260 (4)	3·9 (8)
H(8)	577 (1)	934 (2)	89 (4)	3·3 (8)
H(9)	670 (2)	981 (2)	110 (4)	4·8 (9)
H(10)	698 (2)	1025 (2)	323 (3)	3·1 (7)
H(11)	641 (2)	903 (2)	891 (4)	4·6 (9)
H(12)	633 (2)	984 (3)	841 (4)	5·4 (10)
H(13)	696 (2)	952 (2)	869 (4)	5·7 (11)
H(14)	701 (2)	839 (2)	602 (5)	5·3 (11)
H(15)	688 (2)	814 (3)	741 (5)	6·9 (13)
H(16)	742 (2)	867 (2)	700 (4)	4·2 (8)
H(17)	459 (2)	910 (2)	543 (4)	5·3 (12)

* Parameter held constant during refinement.

A single crystal of approximate dimensions 0·2 × 0·2 × 0·2 mm [μ(Mo $K\alpha$) = 0·47 cm^{-1}] was used. The diffraction conditions hkl: $h + k$, $k + l$, $(l + h) = 2n$, $h0l$: $h + l = 4n$, $0kl$: $k + l = 4n$, the absence of other unrelated conditions, and the orthorhombic symmetry establish the space group as *Fdd*2 (No. 43). Diffraction data were measured at 23 °C with a Picker FACS-I automatic diffractometer using Zr-filtered Mo $K\alpha$ radiation. The cell parameters were determined by a least-squares fit to the angular settings $(2\theta,\omega,\chi,\varphi)$ of 12 reflections in the range $35° \leq 2\theta \leq 40°$ for which the α_1,α_2 doublet was clearly resolved [λ(Mo $K\alpha_1$) = 0·70926 Å]. The 1371 unique reflections (including 256 'unobserved') in the $-h+k+l$ octant were collected for $2\theta \leq 55°$ using the θ–2θ scan method [scan speed 1° (2θ) min^{-1}; 10 s backgrounds; scan range of 1·0° (2θ) plus the α_1–α_2 divergence; and three standard reflections measured after every 100 data were used to scale the data]. The data were reduced and standard deviations calculated as a function of counting statistics as reported previously (Wei & Ward, 1976); the least-squares-refinement weights were calculated from the standard deviations of the structure factors by $w =$

$1/[\sigma^2 + (0{\cdot}02F)^2]$; extinction and absorption corrections were not applied to the data.

The crystal structure was solved using the program *MULTAN* (Germain, Main & Woolfson, 1971). Other programs used in this study include *ORTEP* (Johnson, 1965), the entire system of Zalkin's (1974) programs, and programs written and/or modified locally. A CDC 6500 computer was used.

The structure was refined by full-matrix least-squares to $R_1 = (\sum |F_o - F_c|)/\sum F_o = 0{\cdot}050$, $R_2 = \{[\sum w(F_o - F_c)^2]/\sum w(F_o)^2\}^{1/2} = 0{\cdot}038$; $R_1 = 0{\cdot}072$ including the 256 zero-weighted data for which $I \leq \sigma(I)$, the standard deviation of an observation of unit weight = 1·015; the final atomic parameters are listed in Table 1. The average and maximum shift-to-error ratios for the last cycle of least-squares refinement were 0·072 and 0·257 for non-hydrogen and 0·148 and 0·508 for H atoms. The final difference map showed densities ranging from +0·07 to −0·07 e Å^{-3} with no indication of incorrectly placed or missing atoms. The scattering factors of Doyle & Turner (1968) were used for the non-hydrogen atoms, those of Stewart, Davidson & Simpson (1965) for H; the anomalous scattering factors of Cromer & Liberman (1970) were used for the non-hydrogen atoms, and anomalous scattering factors of zero were assumed for H.* Interatomic distances and angles are listed in Table 2. Refinement of the opposite enantiomorph led to *R* factors and (to within $\frac{1}{4}\sigma$) atomic coordinates identical with those reported above.

Table 2. *Interatomic distances* (Å) *and angles* (°)

The superscript *a* represents an atom at 1 − *x*, 2 − *y*, *z*.

C(1)–C(2)	1·539 (4)	C(2^a)–C(2)–C(3)	110·7 (2)
C(1)–C(8)	1·527 (4)	C(2^a)–C(2)–O(16)	110·6 (3)
C(1)–O(9)	1·432 (4)	C(3)–C(2)–O(16)	107·0 (2)
C(2)–C(2^a)	1·598 (6)	C(2)–C(3)–C(4)	119·5 (3)
C(2)–C(3)	1·520 (4)	C(2)–C(3)–C(10)	121·1 (3)
C(2)–O(16)	1·422 (4)	C(4)–C(3)–C(10)	119·3 (3)
C(3)–C(4)	1·396 (4)	C(3)–C(4)–C(5)	122·0 (3)
C(3)–C(10)	1·395 (5)	C(3)–C(4)–C(13)	118·4 (3)
C(4)–C(5)	1·505 (5)	C(5)–C(4)–C(13)	119·6 (3)
C(4)–C(13)	1·396 (5)	C(4)–C(5)–C(6)	114·9 (3)
C(5)–C(6)	1·528 (5)	C(4)–C(5)–O(9)	110·7 (3)
C(5)–O(9)	1·438 (4)	C(6)–C(5)–O(9)	108·2 (3)
C(6)–C(7)	1·539 (5)	C(5)–C(6)–C(7)	112·7 (3)
C(7)–C(8)	1·539 (4)	C(6)–C(7)–C(8)	108·5 (3)
C(7)–C(14)	1·527 (5)	C(6)–C(7)–C(14)	111·4 (4)
C(7)–C(15)	1·523 (5)	C(6)–C(7)–C(15)	109·5 (4)
C(10)–C(11)	1·381 (5)	C(8)–C(7)–C(14)	109·9 (3)
C(11)–C(12)	1·375 (6)	C(8)–C(7)–C(15)	109·7 (3)
C(12)–C(13)	1·361 (6)	C(14)–C(7)–C(15)	107·8 (4)
C(2)–C(1)–C(8)	113·2 (3)	C(7)–C(8)–C(1)	112·8 (3)
C(2)–C(1)–O(9)	106·3 (3)	C(1)–O(9)–C(5)	109·1 (3)
C(8)–C(1)–O(9)	112·0 (2)	C(3)–C(10)–C(11)	120·4 (3)
C(1)–C(2)–C(2^a)	112·5 (2)	C(10)–C(11)–C(12)	120·3 (3)
C(1)–C(2)–C(3)	106·8 (3)	C(11)–C(12)–C(13)	119·5 (4)
C(1)–C(2)–O(16)	109·1 (2)	C(12)–C(13)–C(4)	122·0 (4)

Hydrogen bonding	H···O	O···O	O–H···O
O(16)–H(17)···O(9^a)	1·91 (5)	2·730	151

Discussion. The structure and the numbering of the atoms of the title compound are shown in Fig. 1. Fig. 2, viewed approximately normal to the twofold axis, shows the joining of the two halves of the molecule by the single C–C bond and by the two hydrogen bonds. The C(2)–C(2′) bond is slightly long at 1·598 (6) Å; the hydrogen bonds appear normal with O(16)···O(9′) = 2·731, H(17)···O(9′) = 1·91 (5) Å, and O(16)–H(17)···O(9′) = 151°. Crowding between the two halves of the molecule, as shown by the slightly long C(2)–C(2′) bond, is also indicated by short intramolecular contacts [H(1)–H(1′) 1·94, C(1)–C(1′) 3·053, C(3)–C(3′) 3·112, C(10)–C(10′) 3·003, C(1)–O(16′) 2·414 and C(3)–O(16′) 2·364 Å], and by the displacement of the benzylic C atoms from the plane of

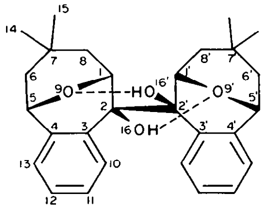

Fig. 1. The molecular structure and the numbering of the atoms.

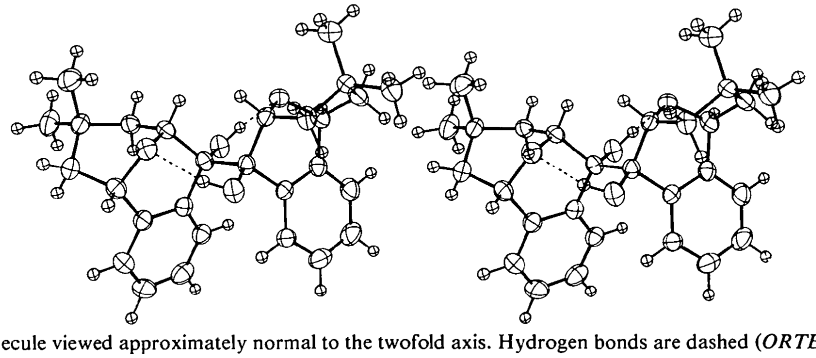

Fig. 2. Single molecule viewed approximately normal to the twofold axis. Hydrogen bonds are dashed (*ORTEP*, Johnson, 1965).

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* Tables of structure factors, anisotropic thermal parameters, torsion angles, and distances and angles involving hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33282 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

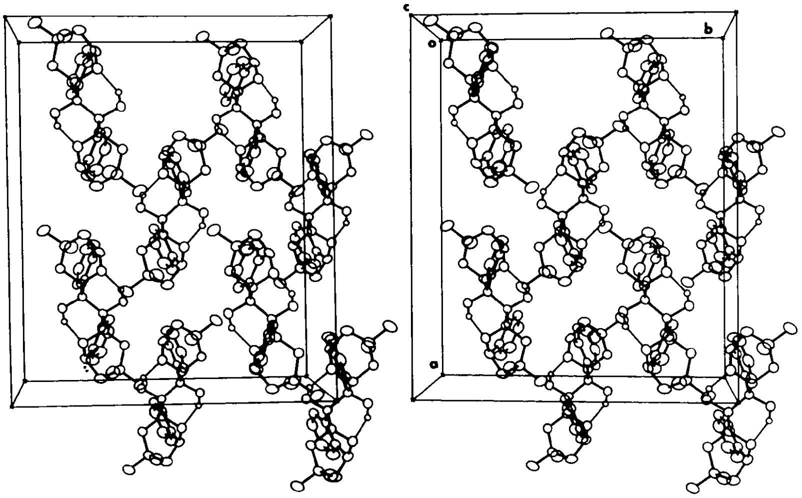

Fig. 3. Packing diagram viewed normal to the *ab* plane (*ORTEP*, Johnson, 1965).

the phenyl ring in the direction away from the other phenyl ring [C(2) −0·03, C(5) −0·10 Å]. The bond distances and angles within the half molecule appear normal except for the C—C—C angles across C(1) and C(5) which average 114·1°; these are the bridgehead atoms in the tricyclic ring system. The ring formed by atoms C(1), C(5)—C(8), and O(9) is in the 'boat' form; an examination of models indicates that this form is preferable to the 'chair' form in which one of the methyl groups would interact strongly with the phenyl ring. The packing of the molecules is shown in Fig. 3. There are no indications of intermolecular hydrogen bonds.

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Structure of the Racemic Pair of Lactones of (2*S*,3*S*)-4-Hydroxyvaline and its Optical Isomer (as their Hydrochlorides)

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Abstract. $C_5H_9NO_2.HCl$, monoclinic, $C2/c$, $a = 16{\cdot}536\,(10)$, $b = 5{\cdot}656\,(10)$, $c = 15{\cdot}68\,(1)$ Å, $\beta = 105{\cdot}2\,(1)°$, $D_m = 1{\cdot}40$, $D_c = 1{\cdot}42$ g cm^{-3} for $Z = 8$. The methyl and ammonium substituents on the lactone ring are *trans*; the ring itself is puckered although four of the ring atoms are coplanar with the O atom of the carbonyl group.

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