The low temperature was obtained with an MSC low-temperature apparatus; the stated temperature, 223 K, is accurate to ±2 K. Scan widths were $(1.60 + 0.35\tan\theta)^\circ$ in ω, with background/scan time-ratio of 0.5. The data were corrected for Lorentz and polarization effects. The intensity decays were treated as linear. As decay at 296 K was so large (very likely owing principally to sublimation), the 223 K study was performed. Ultimately, however, both structures proved to be fully satisfactory. The Laue group assignments, systematic absences and centrosymmetric intensity statistics indicated space group *C*2/*c* (No. 15) for both sets of data; since the refinements proceeded well, it was adopted. Difference Fourier methods were used to locate the initial H-atom positons and the H atoms were then refined isotropically. Subsequently, the H atoms were made canonical, with C—H = 0.98 Å and $U_{\rm iso} = 1.2 \times U_{\rm eq}$ of the attached C atom. Extinction coefficients, which were predicted to be negative in the later least-squares cycles, were not included in the models. The maximum positive residual peaks are located ~0.8 and 1.2 Å, respectively, from C12; similarly, the maximum negative peaks are located ~1.1 and 1.4 Å from C14.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *TEXSAN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

I acknowledge with pleasure my use of the departmental X-ray crystallographic facility, which is supervised by staff crystallographer Dr J. C. Galluci. The diffractometer was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1414). Services for accessing these data are described at the back of the journal.

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6,6′-Dimethoxy-3,3′,5,5′-tetramethylbiphenyl-2,2′-dimethanol

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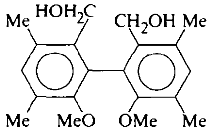
Abstract

The title compound, $C_{20}H_{26}O_4$, crystallized in the centrosymmetric space group $P2_1/c$ with a single molecule as the asymmetric unit. The two hydroxyl-H atoms are ordered and are involved in hydrogen bonds whose $O_D\cdots O_A$ distances are 2.807 (2) and 2.805 (2) Å; one of these bonds is intramolecular. The intramolecular dihedral angle between the ring planes (twist angle) is 87.3 (1)°.

Comment

This report on 6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl-2,2′-dimethanol, (I), is one of a series on hydrogen bonding in substituted biphenyls which includes, most recently, 6,6′-dimethylbiphenyl-2,2′-dicarboxylic acid (Gerkin, 1998). The title compound crystallized in the centrosymmetric space group $P2_1/c$ with a single molecule as the asymmetric unit. The refined molecule is shown in Fig. 1 together with the numbering scheme. Each of the hydroxyl-H atoms is ordered and is involved in a hydrogen bond. Geometric details of these two hydrogen bonds are given in Table 2. Hydrogen-bond graph-set analysis (Bernstein *et al.*, 1995) gives, in the order of Table 2, first-level graphs *C*(9), a chain propagating in the *c* direction which links molecules of space group symmetry types 1 and 4 or 2 and 3, and *S*(9), an intramolecular ring; the basic second-level set is $C_2^2(4)[S(9)]$, a chain of rings with the chain propagating in the *c* direction. These hydrogen bonds are shown in Fig. 2.



(I)

Since the O atoms of the methoxy groups (O3 and O4) are not involved with the hydroxyl-H atoms, significant C—H···O interactions involving them may be expected. Three such interactions having $H\cdots O_A$ dis-

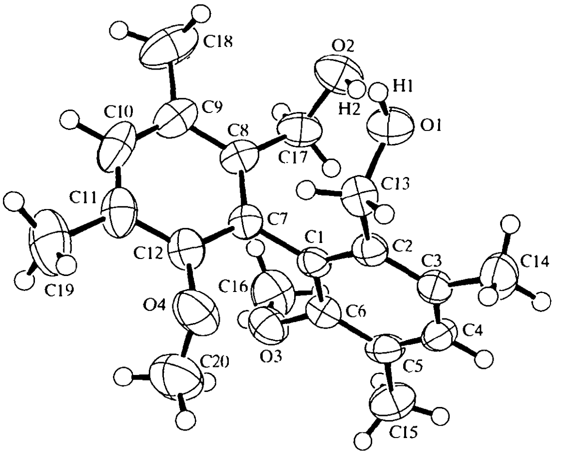


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (I) showing the numbering scheme. Displacement ellipsoids are drawn for 50% probability for all for non-H atoms; circles of arbitrary small radius depict H atoms.

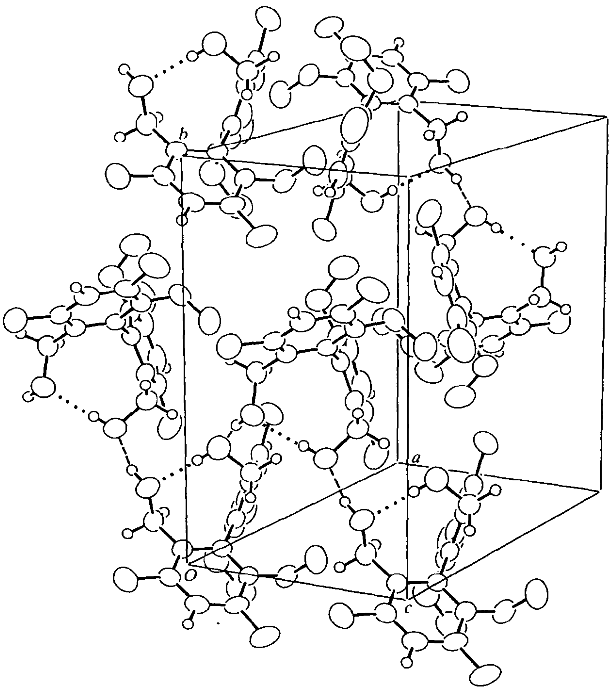


Fig. 2. *ORTEP*II (Johnson, 1976) packing diagram of (I). Displacement ellipsoids are drawn for 50% probability for all for non-H atoms; circles of arbitrary small radius depict H atoms. Four intermolecular hydrogen bonds are shown as dashed lines, with intramolecular hydrogen bonds as dotted lines. Methyl-group-H atoms have been omitted for clarity. The cell origin is at the lower left rear corner.

tances less than the sum of their van der Waals radii (Bondi, 1964) do occur. Of these three, two are intramolecular. Their principal effect is, most probably, to determine the orientation of the methyl-H atoms which participate. Geometric details are given in Table 2.

In this structure, the benzene rings are nearly planar; the maximum deviations of the ring-C atoms from the best-fit planes describing them are 0.011 (2) and 0.014 (2) Å, while the average deviations are 0.007 (3) and 0.008 (4) Å for the C1–C6 and C7–C12 rings, respectively. The intramolecular dihedral angle between these ring planes, the so-called biphenyl twist angle, is 87.3 (1)°. The dihedral angle between the planes of the C1–C6 rings of the two non-parallel sets of molecules is 75.9 (1)°.

For comparison of the biphenyl twist angle and the torsion angle C2—C1—C7—C8, 6,6′-dimethylbiphenyl-2,2′-dicarboxylic acid appears appropriate. In that structure, the biphenyl twist angle is 84.9 (1)° and the torsion angle is of magnitude 88.4 (2)°. In (I), these values are, respectively, 87.3 (1) and 86.5 (3)°. Thus, in these respects, the two molecules are quite similar. Twist angles in biphenyls unsubstituted at the positions *ortho* to the ring–ring bond have been discussed by Brock & Minton (1989).

Distances and angles of special interest in the title molecule are given in Table 1; all distances and angles apparently fall within normal ranges. The closest intermolecular approaches in this structure, excluding pairs of atoms in groups hydrogen bonded to each other, are between C10 and H18C^{i} [symmetry code: (i) $x, \frac{1}{2}-y, z-\frac{1}{2}$] and are 0.28 Å less than the sum of the corresponding van der Waals radii (Bondi, 1964). The molecules in which these atoms occur are, however, hydrogen bonded to each other.

Experimental

The experimental sample of 6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl-2,2′-dimethanol was obtained from the chemical collection of Dr M. S. Newman. The synthesis is described by Newman & Dali (1977).

Crystal data

$C_{20}H_{26}O_4$
$M_r = 330.42$
Monoclinic
$P2_1/c$
$a = 15.894\,(1)$ Å
$b = 13.693\,(2)$ Å
$c = 8.442\,(1)$ Å
$\beta = 94.87\,(1)°$
$V = 1830.6\,(4)$ Å^3
$Z = 4$
$D_x = 1.199$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.5–17.1°$
$\mu = 0.082$ mm^{-1}
$T = 296$ K
Prism
0.35 × 0.31 × 0.27 mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4692 measured reflections

$R_{\text{int}} = 0.021$
$\theta_{\text{max}} = 27.56°$
$h = 0 \rightarrow 20$
$k = -17 \rightarrow 17$
$l = -10 \rightarrow 10$

4221 independent reflections
2095 reflections with
$I > 2\sigma_I$

6 standard reflections
every 150 reflections
intensity decay: 4.0%

Refinement

Refinement on F^2
$R(F) = 0.057$
$wR(F^2) = 0.070$
$S = 1.71$
4221 reflections
225 parameters
H atoms: see below
$w = 1/\sigma^2(F^2)$

$(\Delta/\sigma)_{\max} < 0.01$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: none
Scattering factors from
Stewart *et al.* (1965) (H)
and Creagh & McAuley
(1992) (C, O)

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

O1—C13	1.447 (3)	O3—C16	1.419 (3)
O2—C17	1.439 (3)	O4—C12	1.400 (3)
O3—C6	1.394 (2)	O4—C20	1.396 (3)
C6—O3—C16	115.5 (2)	O4—C12—C7	118.5 (2)
C12—O4—C20	115.4 (2)	O4—C12—C11	118.9 (2)
O3—C6—C1	118.6 (2)	O1—C13—C2	110.1 (2)
O3—C6—C5	119.6 (2)	O2—C17—C8	113.5 (2)

Table 2. *Hydrogen-bonding geometry (Å, °) and significant C—H···O interactions in (I) at 296 K*

Note that no uncertainties are given for quantities involving H19*C*, H20*A* and H20*B* since these H atoms are fixed.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.93 (3)	1.91 (3)	2.807 (2)	163 (3)
O2—H2···O1	0.83 (3)	1.97 (3)	2.805 (2)	175 (3)
C20—H20*B*···O3	0.98	2.52	3.400 (3)	150
C19—H19*C*···O4	0.98	2.57	2.866 (3)	97
C20—H20*A*···O3^{ii}	0.98	2.68	3.630 (3)	165

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $x, -\frac{1}{2} - y, z - \frac{1}{2}$.

Scan widths were $(1.40 + 0.35\tan\theta)^\circ$ in ω, with a background/scan time-ratio of 0.5. The data (with h 0 to 10, k 0 to 17, and l −20 to 20) were corrected for Lorentz and polarization effects. A linear decay correction was applied. The Laue group assignment, systematic absences and centrosymmetric intensity statistics indicated space group $P2_1/a$ (No. 14); the cell was then transformed to the standard $P2_1/c$ setting, and since refinement proceeded well this space group was adopted. Difference Fourier methods were used to locate the initial H-atom positions. All H atoms were then refined isotropically. Subsequently, all H atoms except the hydroxyl-H atoms, H1 and H2, were made canonical, with C—H = 0.98 Å and $U_{\rm iso} = 1.2U_{\rm eq}$ of the attached C atom. The hydroxyl-H atoms were refined isotropically; the refined O—H distances are given in Table 2. An extinction coefficient, predicted to be negative in the later stages of refinement, was not included in the model. The maximum positive residual peak is located between C3 and C14, ~0.6 Å from C3, and the maximum negative peak is located ~0.3 Å from C12.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

We acknowledge with pleasure our use of the departmental X-ray crystallographic facility, which is supervised by staff crystallographer Dr J. C. Gallucci. The diffractometer system was purchased with funds provided, in part, by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1410). Services for accessing these data are described at the back of the journal.

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Hydrogen bonding in [(1*R**,7a*S**)-hexahydro-5-oxo-1*H*-pyrrolizinyl-1-yl]acetic acid

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Abstract

The title acid, [(1*R**,7a*S**)-2,3,5,6,7,7a-hexahydro-5-oxo-1*H*-pyrrolizinyl-1-yl]acetic acid ($C_9H_{13}NO_3$), crystallized in the centrosymmetric space group $P\bar{1}$, with one molecule in the asymmetric unit. The single hydrogen bond, which involves the carboxyl group and the