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(*R*,*R*)-(−)-*trans*-2,3-Bis(hydroxydiphenylmethyl)-1,4-dioxaspiro[4.5]decane†

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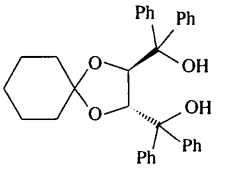
Abstract

The title compound, $C_{34}H_{34}O_4$, contains an intramolecular hydrogen bond involving the hydroxyl group. The crystal packing is stabilized by van der Waals interactions between the hydrophobic surfaces of the neighboring molecules. The observed structure demonstrates novel packing features.

Comment

A host compound based on the 4,5-bis(hydroxydiphenylmethyl)-1,3-dioxolane framework selectively forms an inclusion complex with various guest components such as alcohols, nitriles and esters (for examples, see Mori & Toda, 1990; Toda, Matsuda & Tanaka, 1991). However, a prediction of optimum host–guest pairing is very difficult since the optical resolution capability is often changed dramatically by a slight difference in the structure of a host and/or a guest. We have carried out a single-crystal X-ray structural analysis of the title compound in order to study the effects of the acetal moiety on the crystal structure.



(I)

The labeling scheme and displacement ellipsoids for the compound are depicted in Fig. 1. The cyclohexyl ring adopts a chair conformation. Each of the four phenyl groups is each essentially planar, all deviations from the least-squares plane being less than 0.01 Å. The two dihedral angles between phenyl groups in the diphenylcarbinol moiety, one attached to C(2) and the other attached to C(3), are 74.8 (2) and 86.7 (2)°, respectively. Two diphenylcarbinol groups are also fixed by an intramolecular hydrogen bond between their hydroxyl groups forming a seven-membered ring. The remaining hydroxyl H atom appears to be involved in a weak intermolecular OH···π contact to a phenyl group. As shown in Fig. 2, van der Waals contacts between hydrophobic groups stabilize the crystal packing. Goldberg *et al.* (1990) reported the crystal structure of 2,2-dimethyl-4,5-bis(hydroxydiphenylmethyl)-1,3-dioxolane, which is a dimeric structure formed through intra- and intermolecular hydrogen bonds. Bond distances and angles in the 4,5-bis(hydroxydiphenylmethyl)-1,3-dioxolane moiety of the title compound are nearly the same as those in the 2,2-dimethyl derivative. However, the corresponding torsion angles differ slightly from each other and this may

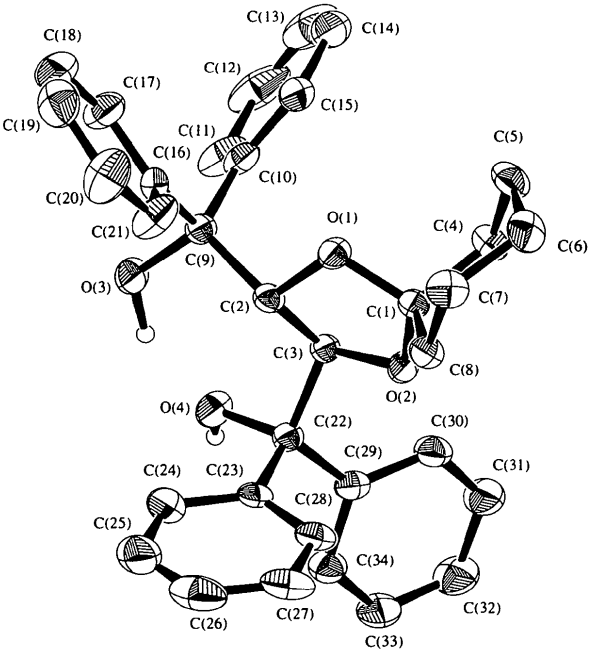


Fig. 1. The labeling scheme and displacement ellipsoids (30% probability) for the title molecule.

† IUPAC name: (*R*,*R*)-(−)-*trans*-1,4-dioxaspiro[4.5]decane-2,3-diylbis(diphenylmethanol).

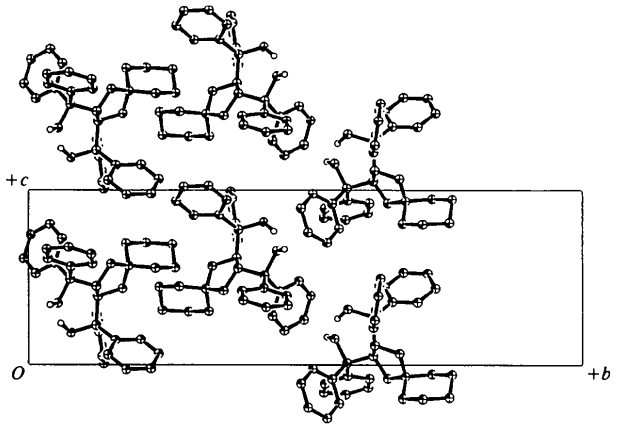


Fig. 2. The crystal structure viewed along the *a* axis showing the hydrogen bonding.

also have an effect on the selectivity of guest molecules. As a result of these differences, the crystal exhibits new packing features.

Experimental

A suitable single crystal of the title compound was obtained by slow recrystallization from *m*-xylene

Crystal data

$C_{34}H_{34}O_4$
M_r = 506.64
Orthorhombic
$P2_12_12_1$
a = 10.210 (7) Å
b = 29.279 (3) Å
c = 9.314 (4) Å
V = 2784 (1) Å^3
Z = 4
D_x = 1.209 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 10.2–14.1°
μ = 0.078 mm^{-1}
T = 296.2 K
Prismatic
0.4 × 0.4 × 0.2 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: analytical (North, Phillips & Mathews, 1968)
T_{min} = 0.972, T_{max} = 0.985
3640 measured reflections
3640 independent reflections

3612 reflections with $I > 0$
θ_{max} = 27.50°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 38$
$l = 0 \rightarrow 12$
3 standard reflections every 150 reflections
intensity decay: −1.43%

Refinement

Refinement on F^2
$R(F)$ = 0.067
$wR(F^2)$ = 0.105
S = 1.109
3612 reflections
344 parameters
H atoms: see below

$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: Zachariasen (1967) type 2 Gaussian isotropic
Extinction coefficient: 0.010 (1)

$w = 1/[\sigma^2(F_o) + 0.00090|F_o|^2]$
$(\Delta/\sigma)_{max} < 0.0001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O(1)—C(1)	1.429 (3)	C(2)—C(3)	1.541 (4)
O(1)—C(2)	1.425 (3)	C(2)—C(9)	1.549 (4)
O(2)—C(1)	1.431 (3)	C(3)—C(22)	1.550 (4)
O(2)—C(3)	1.419 (3)	C(4)—C(5)	1.521 (4)
O(3)—C(9)	1.433 (3)	C(5)—C(6)	1.514 (5)
O(4)—C(22)	1.451 (3)	C(6)—C(7)	1.518 (4)
C(1)—C(4)	1.520 (4)	C(7)—C(8)	1.521 (4)
C(1)—C(8)	1.503 (4)		
C(1)—O(1)—C(2)	108.7 (2)	O(2)—C(3)—C(2)	102.5 (2)
C(1)—O(2)—C(3)	109.6 (2)	O(2)—C(3)—C(22)	107.8 (2)
O(1)—C(1)—O(2)	106.1 (2)	C(2)—C(3)—C(22)	117.2 (2)
O(1)—C(1)—C(4)	108.8 (2)	C(1)—C(4)—C(5)	112.2 (3)
O(1)—C(1)—C(8)	110.9 (2)	C(4)—C(5)—C(6)	110.3 (3)
O(2)—C(1)—C(4)	109.7 (2)	C(5)—C(6)—C(7)	111.1 (3)
O(2)—C(1)—C(8)	108.9 (2)	C(6)—C(7)—C(8)	111.3 (3)
C(4)—C(1)—C(8)	112.1 (2)	C(1)—C(8)—C(7)	111.8 (2)
O(1)—C(2)—C(3)	102.2 (2)	O(3)—C(9)—C(2)	107.5 (2)
O(1)—C(2)—C(9)	107.9 (2)	O(4)—C(22)—C(3)	102.8 (2)
C(3)—C(2)—C(9)	119.6 (3)		

The scan rate was 8° min^{-1} (in ω) and the scan width was (0.84 + 0.30tanθ)°. The ratio of peak-counting time to background-counting time was 2:1. Refinement was carried out by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms. H atoms on the hydroxyl group were located by difference Fourier maps, and all the other H atoms were also located geometrically. H atoms were included but not refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SAPI*91 (Fan, 1991) and *DIRDIF*94 (Beurskens *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1056). Services for accessing these data are described at the back of the journal.

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