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(Z)-3-Benzylidene-1-ethynyl-1-phenyl-1,3-dihydroisobenzofuran

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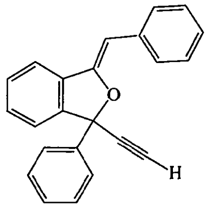
Abstract

The title compound, $C_{23}H_{16}O$, has two independent molecules in the asymmetric unit. The bond lengths and angles of the two molecules are identical. The conformation of the isobenzofuran ring systems of the molecules, however, differ slightly; in molecule *A*, the sp^2-C atom of the five-membered ring deviates the greatest distance [0.034 (2) Å] from the benzo plane, while in molecule *B*, it is the sp^3-C atom of the five-membered ring which has the greatest deviation [0.027 (2) Å] from the benzo plane.

Comment

As part of a structural study involving [3]cumulene intermediates bearing the acetylenic functionality (Ramos & Garcia, 1997), the precursor [1-phenyl-1-(2-phenylethynyl)-3-trimethylsilyl-2-propyn-1-ol] to the title compound was prepared (Ramos & Garcia, 1997). The title compound resulted from an attempt to trimethylsilyl-deprotect the precursor under K_2CO_3 in anhydrous methanol conditions (Austin *et al.*, 1981; Garcia, Ramos, Pratt & Rodriguez, 1995; Garcia, Ramos, Rodriguez & Fronczek, 1995). The title compound, (I), was unexpectedly formed *via* a nucleophilic attack of the hydroxy O atom on the (phenyl)acetylenic functionality, resulting in the formation of the isobenzofuran ring system. Its crystal structure was determined in order to ascertain its identity.



(I)

The title compound exists as two independent molecules in the asymmetric unit, a case which is observed in about 4.6% of compounds having the $P2_1/c$ space group (Brock & Dunitz, 1994). Bond distances and angles of the two molecules are the same within experimental error. The O1*a*—C1*a*, O1*a*—C15*a*, O1*b*—C1*b* and O1*b*—C15*b* bond lengths of 1.385 (2), 1.462 (2), 1.382 (2) and 1.463 (2) Å, respectively, compare well with corresponding lengths in a compound having a similar ring system, *i.e.* 2-isopropyl-4,4,7-trimethyl-1*H*-phenaleno[1,9-*bc*]furan-1-one (Weber *et al.*, 1975). In molecule *A*, the C15 atom lies essentially in the plane of the C9–C14 phenyl ring, deviating from it by only 0.004 (2) Å. The O1*a* and C1*a* atoms, however, deviate by −0.022 (1) and 0.034 (2) Å, respectively, from the same plane. Contrary to C15*a*, C15*b* deviates by −0.027 (2) Å from the plane of the C9–C14 phenyl ring. Atoms O1*b* and C1*b* do not deviate quite as much [0.018 (1) and 0.013 (2) Å, respectively] as C1*a* and O1*a*. Atoms C15*a* and C15*b* lie 0.090 (2) and 0.080 (2) Å out of the C18–C23 phenyl ring planes of their corresponding molecules. The C9*a*—C1*a*—C2*a*—C3*a* torsion angle is 179.5 (2)°, indicating a fairly planar environment about the C1*a*=C2*a* double bond. The C1*b*=C2*b* double bond is less planar, with a C9*b*—C1*b*—C2*b*—C3*b* torsion angle of 178.28 (2)°. The C16*a*≡C17*a* and C16*b*≡C17*b* triple-bond distances of 1.164 (3) and 1.168 (3) Å, respectively, agree with the expected length of 1.174 (4) Å (Allen *et al.*, 1987).

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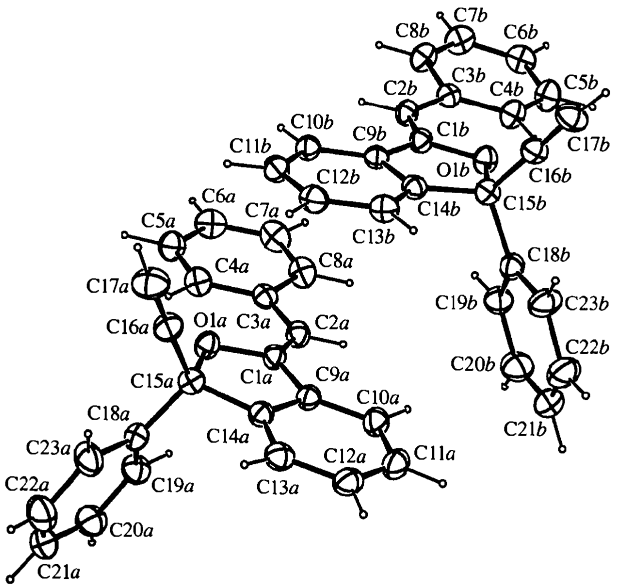

Fig. 1. The two independent molecules in the asymmetric unit with displacement ellipsoids plotted at the 20% probability level.

Experimental

Crystals of the title compound were grown by evaporation of a hexane solution.

Crystal data

$C_{23}H_{16}O$
$M_r = 308.4$
Monoclinic
$P2_1/c$
$a = 10.130\,(1)$ Å
$b = 31.398\,(2)$ Å
$c = 10.7388\,(9)$ Å
$\beta = 90.852\,(7)°$
$V = 3415.2\,(9)$ Å^3
$Z = 8$
$D_x = 1.199$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 15 reflections
$\theta = 19–23°$
$\mu = 0.52$ mm^{-1}
$T = 294$ K
Rectangular prism
0.20 × 0.17 × 0.15 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
7589 measured reflections
7057 independent reflections
4394 reflections with $I > \sigma(I)$

$R_{\rm int} = 0.012$
$\theta_{\rm max} = 75°$
$h = -12 \rightarrow 12$
$k = -39 \rightarrow 0$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.051$
$wR = 0.044$
$S = 1.512$
4394 reflections
562 parameters
All H atoms refined

$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.06$ e Å^{-3}
Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: $7.1\,(3) \times 10^{-7}$

$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$
$(\Delta/\sigma)_{\rm max} = 0.009$

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

O1*a*—C1*a*	1.385 (2)	O1*b*—C1*b*	1.382 (2)
O1*a*—C15*a*	1.462 (2)	O1*b*—C15*b*	1.463 (2)
C1*a*—C2*a*	1.330 (3)	C1*b*—C2*b*	1.332 (3)
C1*a*—C9*a*	1.457 (3)	C1*b*—C9*b*	1.457 (3)
C2*a*—C3*a*	1.449 (3)	C2*b*—C3*b*	1.450 (3)
C9*a*—C14*a*	1.380 (3)	C9*b*—C14*b*	1.381 (3)
C14*a*—C15*a*	1.511 (3)	C14*b*—C15*b*	1.509 (3)
C15*a*—C16*a*	1.473 (3)	C15*b*—C16*b*	1.482 (3)
C15*a*—C18*a*	1.520 (3)	C15*b*—C18*b*	1.523 (3)
C16*a*—C17*a*	1.164 (3)	C16*b*—C17*b*	1.168 (3)
C1*a*—O1*a*—C15*a*	110.8 (1)	C1*b*—O1*b*—C15*b*	110.7 (1)
O1*a*—C15*a*—C14*a*	103.2 (1)	O1*b*—C15*b*—C14*b*	103.2 (1)
O1*a*—C15*a*—C16*a*	107.8 (1)	O1*b*—C15*b*—C16*b*	108.0 (1)
O1*a*—C15*a*—C18*a*	108.5 (1)	O1*b*—C15*b*—C18*b*	108.7 (1)
C14*a*—C15*a*—C16*a*	111.0 (2)	C14*b*—C15*b*—C16*b*	111.5 (2)
C14*a*—C15*a*—C18*a*	112.6 (2)	C14*b*—C15*b*—C18*b*	112.9 (2)
C16*a*—C15*a*—C18*a*	113.1 (2)	C16*b*—C15*b*—C18*b*	111.9 (2)

A series of ψ scans showed no change in intensity with rotation about the diffraction vector. Thus, no absorption corrections were applied. The isotropic displacement parameters ($B_{\rm iso}$) of the H atoms range from 4.8 (4) to 14.1 (8) Å^2 and C—H distances are within the range 0.90 (2) to 1.03 (2) Å.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS MolEN* (Fair, 1990). Program(s) used to solve structure: direct methods *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF IN MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1131). Services for accessing these data are described at the back of the journal.

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

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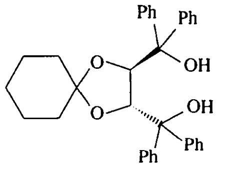
(*Received 5 December 1996; accepted 7 July 1997*)

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

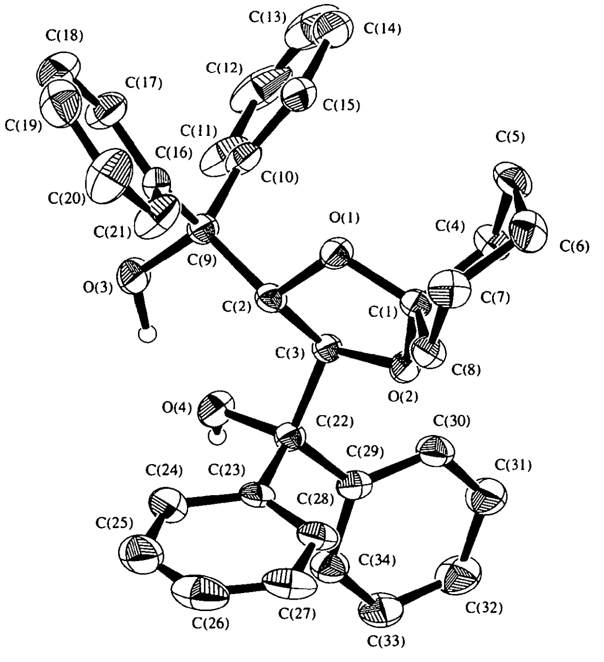


Fig. 1. The labeling scheme and displacement ellipsoids (30% probability) for the title molecule.

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

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