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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.064
wR factor = 0.143
Data-to-parameter ratio = 14.2

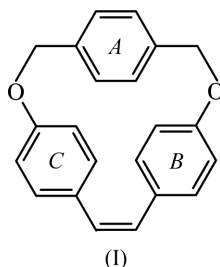
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

7,14-Dioxatetracyclo[14.2^{4,5}.2^{19,20}.2^{21,22}.2^{23,24}]- tetracos-1,3,5,9,11,15,17,19,21,23-decaene

In the title molecule, C₂₂H₁₈O₂, the dihedral angles formed by the planes of the benzene rings are 62.6 (1), 66.1 (1) and 51.4 (1)°. The cavity enclosed by the three benzene rings is approximately 21.5 Å² in area. In addition to van der Waals interactions, the crystal structure is stabilized by weak C—H···π and C—H···O interactions.

Comment

Stilbene-based cyclophanes possess novel optical properties due to their ability to exhibit *cis-trans* isomerization (Tanner & Wennerstrom, 1981). Such a class of cyclophanes, called stilbenophanes, can be obtained by the McMurry coupling technique (Furstner & Bogdanovic, 1996). Treatment of the dialdehyde derived by *o*-alkylation of *p*-hydroxybenzaldehyde with *p*-xylene dibromide with TiCl₄ and Zn in THF afforded the title compound, (I) (Murali, 2002); we report here its crystal structure.



In the title molecule, the benzene rings *A* (C2–C7), *B* (C9–C14) and *C* (C17–C22) are slightly puckered, with *para* C atoms lying on the same side of the plane of the remaining four atoms [−0.048 (2) and −0.037 (2) Å in ring *A*, 0.040 (2) and 0.049 (2) Å in ring *B*, and 0.037 (2) and 0.036 (2) Å in ring *C*]. Similar puckering in the benzene rings of a cyclophane have been reported by Hanson & Rohrl (1972).

The average C—C bond length in the three benzene rings is 1.382 (3) (ring *A*), 1.385 (3) (ring *B*) and 1.382 (3) Å (ring *C*). The average C—C bond length in the benzene rings of the *cis*-stilbene moiety is close to those values [1.378 (11) and 1.380 (9) Å] reported for such rings in an analogue of *cis*-stilbene (Bernstein, 1975). The C15—C16 bond length of 1.317 (4) Å is shorter than the mean value [1.34 Å] reported for analogues of *cis*-stilbene (Bernstein, 1975; Beddoes *et al.*, 1993; Yoon *et al.*, 1995). The widening of the exocyclic bond angle O1—C20—C21 to 124.1 (2)° and consequent narrowing of O1—C20—C19 to 116.9 (2)° is due to the close approach [2.21 (3) Å] of atoms H1A and H21, attached to C1 and C21, respectively. Similarly, the widening of the bond angle O2—

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C9–C14 to 124.8 (2)° may be due to the close approach [2.23 (3) Å] of atoms H8A and H14, attached to C8 and C14, respectively. The O–C–C angles at the sp^3 hybridized atoms, C1 [O1–C1–C2 = 111.6 (2)°] and C8 [O2–C8–C5 = 111.7 (2)°], deviate somewhat from the ideal tetrahedral value.

The weighted least-squares plane through ring *A* makes dihedral angles of 62.6 (1) and 66.1 (1)°, respectively, with the planes through rings *B* and *C*. The dihedral angle between the mean planes of the two benzene rings (*B* and *C*) of the *cis*-stilbene moiety is 51.4 (1)°, compared to 62° in 1,2-diphenylcyclopentene (Bernstein, 1975), and 62.1 (4) (molecule *A*) and 52.7 (4)° (molecule *B*) in 2,3-diphenylmaleic anhydride (Yoon *et al.*, 1995). The torsion angle C12–C15–C16–C17 of –7.0 (4)° is close to the value of –7.5° for 1,2-diphenylcyclopentene (Bernstein, 1975) and to the theoretically calculated value of –9° for *cis*-stilbene (Bernstein, 1975). The size of the cavity enclosed by the three benzene rings can be approximately measured as the area enclosed by C1···C8···C15–C16···C1, 21.5 Å².

In addition to van der Waals interactions, the crystal structure is stabilized by weak C–H···O and C–H··· π interactions involving screw- and glide-related molecules (Table 2).

Experimental

Reaction of *p*-xylylenyl dibromide with *p*-hydroxybenzaldehyde in anhydrous DMF and in the presence of K₂CO₃ at 343 K for 48 h gave *p*-bis(*p*-formylphenoxy)methylbenzene. Treatment of *p*-bis(*p*-formylphenoxy)methylbenzene with 5 equivalents of TiCl₄ and 10 equivalents of Zn in dry THF for 24 h under high dilution conditions afforded the title compound in 53% yield. It was purified by column chromatography (SiO₂; hexane–chloroform = 8:2) and single crystals suitable for X-ray study were grown by vapor diffusion of hexane into a solution of the title compound in chloroform.

Crystal data

C ₂₂ H ₁₈ O ₂	$D_x = 1.239 \text{ Mg m}^{-3}$
$M_r = 314.36$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4210 reflections
$a = 9.5687$ (2) Å	$\theta = 2.6\text{--}28.3^\circ$
$b = 10.5926$ (2) Å	$\mu = 0.08 \text{ mm}^{-1}$
$c = 16.6960$ (2) Å	$T = 293$ (2) K
$\beta = 95.423$ (1)°	Block, colourless
$V = 1684.69$ (5) Å ³	$0.42 \times 0.26 \times 0.16 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	1784 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.114$
Absorption correction: none	$\theta_{\text{max}} = 28.3^\circ$
9778 measured reflections	$h = -12 \rightarrow 12$
4108 independent reflections	$k = -14 \rightarrow 13$
	$l = -22 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.015P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.064$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.143$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.82$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
4107 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
290 parameters	Extinction correction: <i>SHELXTL</i>
All H-atom parameters refined	Extinction coefficient: 0.044 (4)

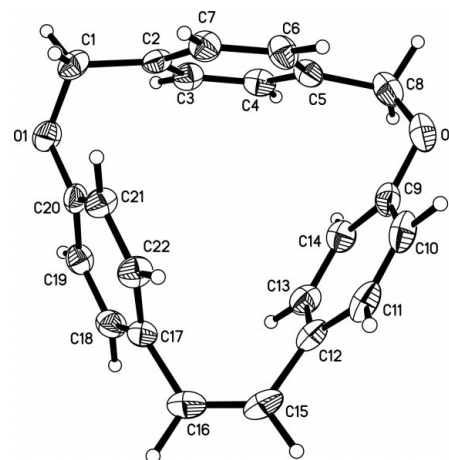


Figure 1

The structure of the title compound, with the atom-numbering scheme, and displacement ellipsoids drawn at the 30% probability level.

Table 1

Selected geometric parameters (Å, °).

O1–C20	1.379 (3)	C5–C8	1.517 (3)
O1–C1	1.440 (3)	C12–C15	1.476 (4)
O2–C9	1.372 (3)	C15–C16	1.317 (4)
O2–C8	1.441 (3)	C16–C17	1.486 (3)
C1–C2	1.504 (3)		
C20–O1–C1	116.77 (16)	C11–C12–C13	116.7 (2)
C9–O2–C8	117.23 (19)	C11–C12–C15	122.5 (2)
O1–C1–C2	111.59 (19)	C22–C17–C18	117.1 (2)
O2–C8–C5	111.7 (2)	C18–C17–C16	123.0 (2)
O2–C9–C10	116.2 (2)	O1–C20–C19	116.9 (2)
O2–C9–C14	124.8 (2)	O1–C20–C21	124.1 (2)
C12–C15–C16–C17	–7.0 (4)		

Table 2

Hydrogen-bonding geometry (Å, °).

C_gB and C_gC denote the centroids of phenyl rings *B* and *C*, respectively.

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C22–H22···O2 ⁱ	0.99 (2)	2.58 (2)	3.438 (3)	145 (2)
C8–H8B···C _g C ⁱⁱ	1.03 (2)	2.75 (2)	3.715 (3)	157 (2)
C7–H7···C _g B ⁱ	1.02 (2)	3.01 (2)	3.841 (3)	140 (1)

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

H atoms were located from a difference map and both positional and isotropic displacement parameters were refined. For H atoms, the C–H range is 0.95 (2)–1.05 (3) Å and the U_{iso} range is 0.036 (5)–0.102 (10) Å². The reflection (120) was removed during refinement, as the observed and calculated structure factors showed large disagreement. The high R_{int} value (0.114) and low ratio of observed to unique reflections (0.43) may be a result of the poor diffraction quality of the crystal.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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