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Key indicators

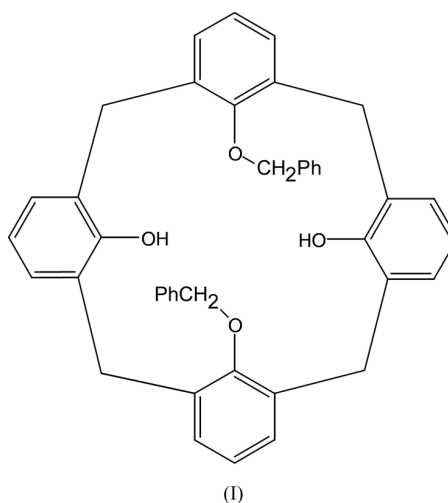
Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.060
 wR factor = 0.134
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.25,27-Bis(benzyloxy)-26,28-dihydroxy-
calix[4]areneThe title compound, $\text{C}_{42}\text{H}_{36}\text{O}_4$, belongs to the class of calix[4]-
arenes. It assumes a cone conformation. The hydroxy groups
form intramolecular hydrogen bonds to the ether O atoms.

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Comment

Calixarenes are enjoying considerable interest in the field of
supramolecular chemistry because their derivatives can form
inclusion complexes with cations or with neutral molecules
(Gutsche, 1989; Vicens & Böhmer, 1991).

The molecular structure of the title compound, (I), is shown in Fig. 1. The calix[4]arene assumes a conformation with approximate C_2 symmetry in which the C atoms of the methylene bridges are nearly coplanar (average deviation from the mean plane = 0.123 \AA). All four residues (the two hydroxy groups and the two benzyloxy groups) are on the same side of this plane. The aromatic rings of the calix[4]arene form a cone. The interplanar angles of the single aromatic rings with the above-defined mean plane are 72.82 (6), 40.49 (7), 71.02 (5) and 38.76 (5) $^\circ$ for the rings C11–C16, C21–C26, C31–C36 and C41–C46, respectively. The torsion angles around the Ar–CH₂ bonds, which may always be used to give an unambiguous description of the molecular conformation (Ugozzoli & Andreetti, 1992), are given in Table 1. The molecular conformation is stabilized by two intramolecular hydrogen bonds from the hydroxyl groups to the ether O atoms.

Experimental

The title compound, (I), was synthesized according to the procedure described by Casnati *et al.* (1991). Yellow crystals were grown from a methanol/dichloromethane solution.

Crystal data

$C_{42}H_{36}O_4$
 $M_r = 604.71$
 Triclinic, $P\bar{1}$
 $a = 10.3942$ (8) Å
 $b = 12.501$ (1) Å
 $c = 14.348$ (1) Å
 $\alpha = 74.028$ (4)°
 $\beta = 73.954$ (4)°
 $\gamma = 65.501$ (5)°
 $V = 1602.7$ (2) Å³

$Z = 2$
 $D_x = 1.253$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 503
 reflections
 $\theta = 1-25^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 173$ (2) K
 Block, yellow
 $0.43 \times 0.28 \times 0.22$ mm

Data collection

Siemens SMART CCD
 diffractometer
 ω scans
 Absorption correction: none
 30084 measured reflections
 7409 independent reflections

4900 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.042$
 $\theta_{max} = 29.0^\circ$
 $h = -13 \rightarrow 12$
 $k = -16 \rightarrow 16$
 $l = -19 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.134$
 $S = 1.05$
 7409 reflections
 424 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.7201P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.47$ e Å⁻³
 $\Delta\rho_{min} = -0.18$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0109 (13)

Table 1

Selected geometric parameters (Å, °).

C12—O51	1.399 (2)	C42—O42	1.365 (2)
C22—O22	1.360 (2)	O51—C51	1.441 (2)
C32—O61	1.404 (2)	O61—C61	1.448 (2)
C12—O51—C51	113.06 (14)	C32—O61—C61	112.10 (14)
C43—C1—C11—C12	-98.5 (2)	C23—C3—C31—C32	-96.8 (2)
C21—C2—C13—C12	108.9 (2)	C41—C4—C33—C32	106.9 (2)
C13—C2—C21—C22	-81.5 (2)	C33—C4—C41—C42	-80.2 (2)
C31—C3—C23—C22	72.0 (2)	C11—C1—C43—C42	72.0 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O22—H22 \cdots O61	0.86 (3)	1.87 (3)	2.7179 (19)	170 (2)
O42—H42 \cdots O51	0.88 (3)	1.91 (3)	2.768 (2)	162 (2)

H atoms bonded to C atoms were refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$] using a riding model, with C—H = 0.95 and 0.99 Å for aromatic and methylene H atoms, respectively. H atoms bonded to O atoms were refined isotropically.

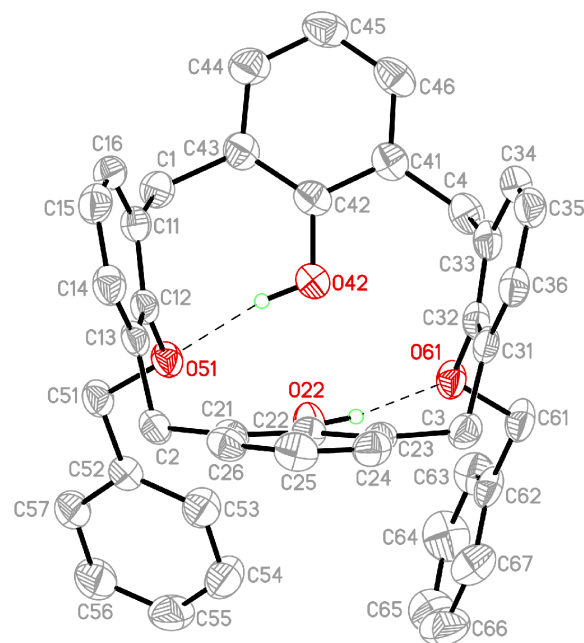


Figure 1

Perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. H atoms bonded to C atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 1990).

References

- Casnati, A., Arduini, A., Ghidini, E., Pochini, A. & Ungaro, R. (1991). *Tetrahedron*, **47**, 2221–2228.
- Gutsche, C. D. (1989). In *Calixarenes, Monographs in Supramolecular Chemistry*, Vol. 1, edited by J. F. Stoddart. London: The Royal Society of Chemistry.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Ugozzoli, F. & Andreotti, G. D. (1992). *J. Incl. Phenom. Mol. Recogn. Chem.* **13**, 337–348.
- Vicens, J. & Böhmer, V. (1991). In *Calixarenes: A Versatile Class of Macrocyclic Compounds*. Dordrecht: Kluwer Academic Publishers.