

A monoclinic polymorph of 25,27-bis(benzyloxy)-
26,28-dihydroxycalix[4]arene

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Key indicators

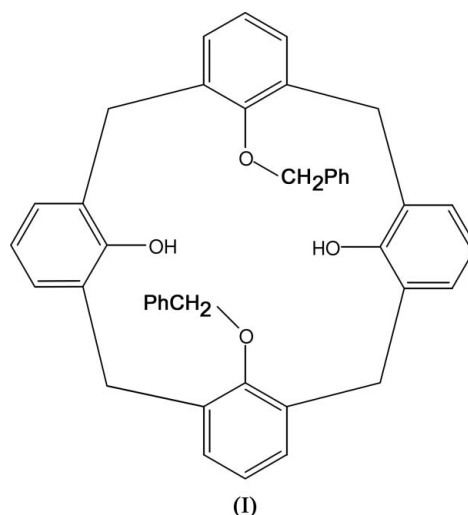
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.053
 wR factor = 0.145
Data-to-parameter ratio = 23.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

A new polymorph of the title compound, $\text{C}_{42}\text{H}_{36}\text{O}_4$, is reported. It crystallizes in space group $P2_1/c$, whereas the previously known polymorph [Bolte & Sakhaii (2004), *Acta Cryst. C60*, o1360–o1361] crystallizes in space group $P\bar{1}$. The conformations of the molecules differ in the orientation of one benzyloxy residue.

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Comment

A triclinic polymorph (space group $P\bar{1}$), (II), of the title compound has already been described (Bolte & Sakhaii, 2004). In this work, a monoclinic polymorph, (I), of the title compound is reported. It crystallizes in space group $P2_1/c$.



A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.0; Allen, 2002).

A least-squares fit (Fig. 2) of the central calixarene framework of (I) and (II) (r.m.s. deviation 0.06 Å) reveals that the two molecules differ only in the orientation of one of the two benzyloxy residues (Table 3).

Two intramolecular hydrogen bonds stabilize the molecular conformation. The different orientations of the benzyloxy residue in (I) and (II) do not affect these hydrogen bonds.

Experimental

The title compound was synthesized according to the procedure described by Casnati *et al.* (1991). Colourless crystals suitable for X-ray diffraction were grown from a methanol–chloroform solution (1:1) of the title compound.

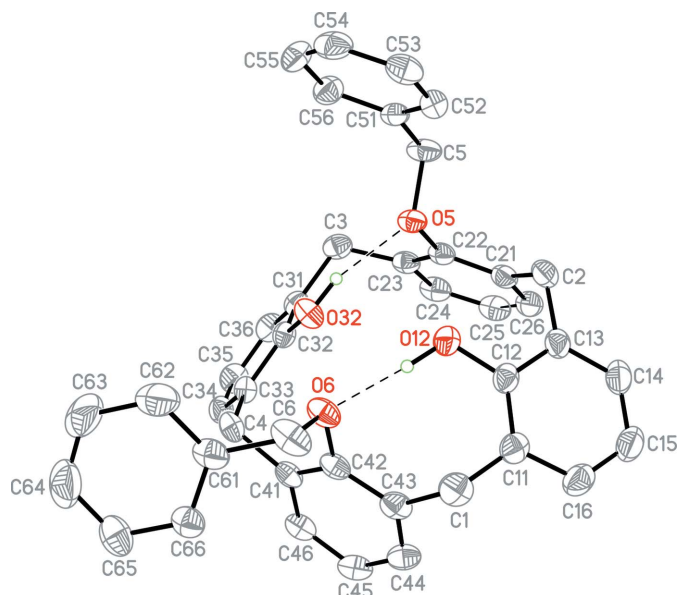


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms bonded to C atoms have been omitted. Dashed lines indicate hydrogen bonds.



Figure 2
A least-squares fit of the two polymorphs of the title compound. Full lines indicate polymorph (I) and dashed lines indicate polymorph (II). H atoms have been omitted.

Crystal data

$C_{42}H_{36}O_4$
 $M_r = 604.71$
 Monoclinic, $P2_1/c$
 $a = 11.250 (1) \text{ \AA}$
 $b = 15.380 (1) \text{ \AA}$
 $c = 18.616 (2) \text{ \AA}$
 $\beta = 95.31 (1)^\circ$
 $V = 3207.2 (5) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.252 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 8192 reflections
 $\theta = 3.5\text{--}25.0^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Block, colourless
 $0.50 \times 0.50 \times 0.30 \text{ mm}$

Data collection

Siemens SMART CCD three-circle diffractometer
 ω scans
 Absorption correction: none
 27936 measured reflections
 9849 independent reflections

5829 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.046$
 $\theta_{max} = 31.9^\circ$
 $h = -15 \rightarrow 16$
 $k = -23 \rightarrow 22$
 $l = -27 \rightarrow 27$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.145$
 $S = 1.01$
 9849 reflections
 423 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 6.09P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.20 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (\AA).

O5—C22	1.4046 (15)	O6—C6	1.4671 (18)
O5—C5	1.4516 (17)	C12—O12	1.3668 (17)
O6—C42	1.3974 (17)	O32—C32	1.3650 (16)

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O12—H12 \cdots O6	0.96 (2)	1.85 (2)	2.8077 (15)	173 (2)
O32—H32 \cdots O5	0.90 (2)	1.93 (2)	2.8056 (14)	163.7 (19)

Table 3

Comparison of torsion angles ($^\circ$) for the two polymorphs, (I) and (II).

	(I)	(II)
C42—O6—C6—C61	75.91 (18)	−160.16 (16)
O6—C6—C61—C62	−88.8 (2)	−166.37 (18)
O6—C6—C61—C66	88.20 (19)	14.4 (3)
C41—C42—O6—C6	99.91 (15)	87.4 (2)
C43—C42—O6—C6	−83.46 (16)	−95.22 (19)

H atoms were located in a difference map, but refined with fixed individual isotropic displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$] using a riding model, with C—H = 0.93 and 0.97 \AA , for $C_{aromatic}$ and $C_{methylene}$, respectively. The hydroxyl H atoms were refined isotropically.

Data collection: SMART (Siemens, 1995); cell refinement: SAINT (Siemens, 1995); data reduction: SAINT; 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.

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