

5,11,17,23-Tetra-*tert*-butyl-25,26,27,28-tetra-pentoxycalix[4]areneVasily Brusko,^a Volker Böhmer^a
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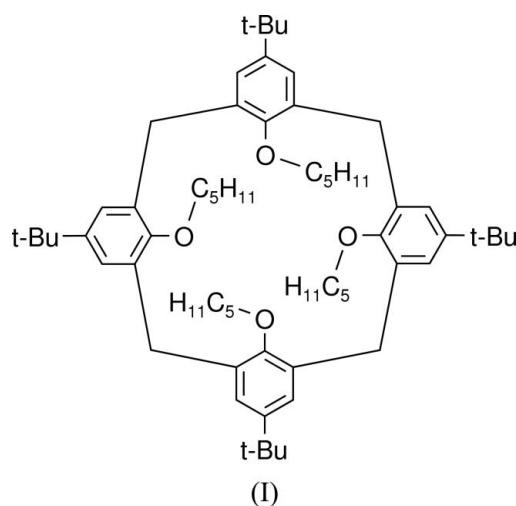
Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.050
 wR factor = 0.148
Data-to-parameter ratio = 18.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound, $\text{C}_{64}\text{H}_{96}\text{O}_4$, adopts the typical pinched-cone conformation. The dihedral angles between the reference plane (defined by the C atoms of the methylene bridges) and the benzene rings are 86.88 (4), 136.64 (5), 87.22 (4) and 133.99 (4)°.

Comment

A perspective view of the title compound, (I), is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL*, Version 1.0; Allen, 2002). The molecule adopts the typical pinched-cone conformation. The reference plane of the calixarene, defined as the mean plane of the bridging C atoms (here C1, C2, C3 and C4) is almost perfectly planar (r.m.s. deviation = 0.0817 Å); rings C11–C16 and C31–C36 subtend angles of 86.88 (4) and 87.22 (4)°, respectively, with this plane, whereas rings C21–C26 and C41–C46 enclose angles of 136.64 (5) and 133.99 (4)°, respectively, with this plane. Thus, the former rings are slightly bent inwards, whereas the others are clearly bent outwards. Rings C11–C16 and C31–C36 are almost coplanar [0.40 (11)°], whereas the other two are almost perpendicular to each other [89.37 (5)°]. The torsion angles describing the orientation of the aromatic rings with respect to the reference plane are listed in Table 1. Two pentoxy chains adopt all-*trans* conformations, whereas in the remaining two chains *gauche* torsion angles are found.

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Experimental

The title tetrapentyl ether, (I), was prepared by *O*-alkylation of *tert*-butylcalix[4]arene with pentyl bromide under standard conditions (Jakobi *et al.*, 1996). Crystals were obtained from a chloroform solution.

Crystal data

$C_{64}H_{96}O_4$
 $M_r = 929.41$
 Monoclinic, $P2_1/n$
 $a = 15.8573$ (8) Å
 $b = 20.0393$ (13) Å
 $c = 20.0147$ (10) Å
 $\beta = 110.436$ (4)°
 $V = 5959.8$ (6) Å³
 $Z = 4$

$D_x = 1.036$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 77341
 reflections
 $\theta = 2.0$ – 25.8 °
 $\mu = 0.06$ mm⁻¹
 $T = 173$ (2) K
 Block, colourless
 $0.46 \times 0.42 \times 0.24$ mm

Data collection

Stoe IPDS-II two-circle
 diffractometer
 ω scans
 Absorption correction: none
 84719 measured reflections
 11556 independent reflections

8021 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.071$
 $\theta_{max} = 25.9$ °
 $h = -19 \rightarrow 19$
 $k = -24 \rightarrow 24$
 $l = -24 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.148$
 $S = 1.04$
 11556 reflections
 632 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0906P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.33$ e Å⁻³
 $\Delta\rho_{min} = -0.17$ e Å⁻³

Table 1

Selected torsion angles (°).

C43–C1–C11–C12	114.88 (16)	C23–C3–C31–C32	118.52 (16)
C12–C13–C2–C21	-115.35 (16)	C32–C33–C4–C41	-118.75 (16)
C13–C2–C21–C22	75.08 (19)	C33–C4–C41–C42	76.96 (19)
C22–C23–C3–C31	-72.3 (2)	C11–C1–C43–C42	-76.37 (18)

H atoms were located in a difference electron-density map, but refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl C})$] using a riding model, with C–H distances ranging from 0.95 to 0.99 Å. Two C atoms of one pentoxy chain are disordered over two sites, with occupation factors of 0.520 (6) and 0.480 (6). The bonds involving the disordered atoms were refined with a distance restraint of 1.54 (1) Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in

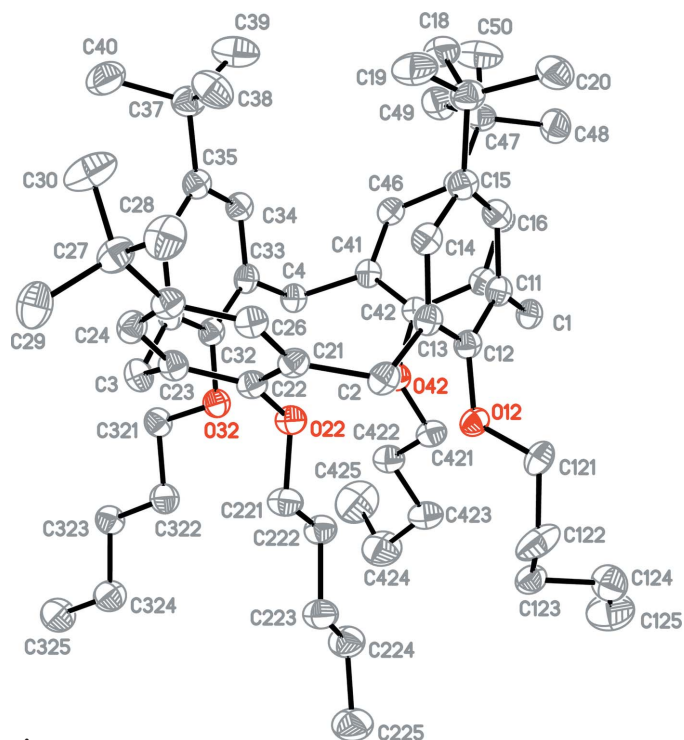


Figure 1

A perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 30% probability level. Only one component of the disordered pentoxy chain is shown; C12', C12'' and H atoms have been omitted for clarity.

SHELXTL-Plus (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Jakobi, R. A., Böhmer, V., Grüttner, C., Kraft, D. & Vogt, W. (1996). *New J. Chem.* **20**, 493–501.
 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.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
 Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.