Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,17-Di-*tert*-butyl-25,27-dihydroxycalix[4]arene

Jeffrey C. Bryan,* Richard A. Sachleben and Agathe Urvoas

Chemical and Analytical Sciences Division,
Oak Ridge National Laboratory, Oak Ridge,
TN 37831-6119, USA

Correspondence e-mail: bryanjc@ornl.gov

Received 28 June 2001
Accepted 18 July 2001
Online 27 July 2001

Half of the title compound, $C_{38}H_{44}O_2$, makes up the asymmetric unit; the whole molecule possesses inversion symmetry. The calix[4]arene conformation is similar to the unsubstituted calix[4]arene. Only one intermolecular interaction is noted.

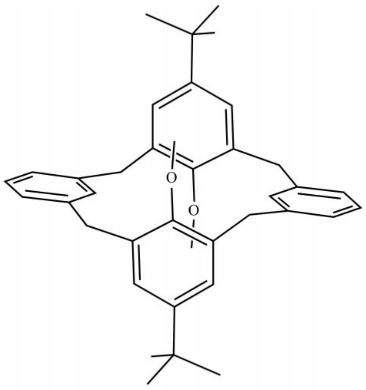
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.108
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.

Comment

The title compound was prepared as part of our investigations into the synthesis of new calix[4]arene-based crown ether extractants (Sachleben *et al.*, 1999). Only half of the molecule makes up the asymmetric unit; the other half is generated by the crystallographic inversion centre [symmetry code:$1 - x, 1 - y, 1 - z$, denoted (i) throughout this paper]. The conformation of a calix[4]arene is generally defined in terms of the relative orientation of the arene rings with respect to the best plane defined by the four calix methylene-C atoms (C17, C18, C17^{i}, and C18^{i}). This methylene carbon plane is referred to as the reference plane (Perrin & Oehler, 1991). The planes of the two methoxy-substituted arene rings (A1 and $A1^{i}$) are nearly perpendicular to the reference plane [84.5 (1)°], while the unsubstituted arene rings (A2 and $A2^{i}$) appear to be roughly parallel to it [173.9 (1)°]. This calix configuration closely resembles that of the unsubstituted parent molecule, calix[4]arene (McMurry & Phelan, 1991) and that of tetra-*tert*-butyl-calix[4]arene (Grynszpan *et al.*, 1991). Strangely, the more closely related tetra-*tert*-butyl-diethoxy-calix[4]arene adopts a 1,3-alternate calix conformation (Ting *et al.*, 1995).



(I)

Examination of the extended structure reveals only one intermolecular interaction worthy of mention, an edge–face arene interaction between A2 and A1. The H16$\cdots A1_{\rm centroid}$ distance is 2.64 Å and the C16—H16$\cdots A1_{\rm centroid}$ angle is 160°.

DOI: 10.1107/S160053680101220X

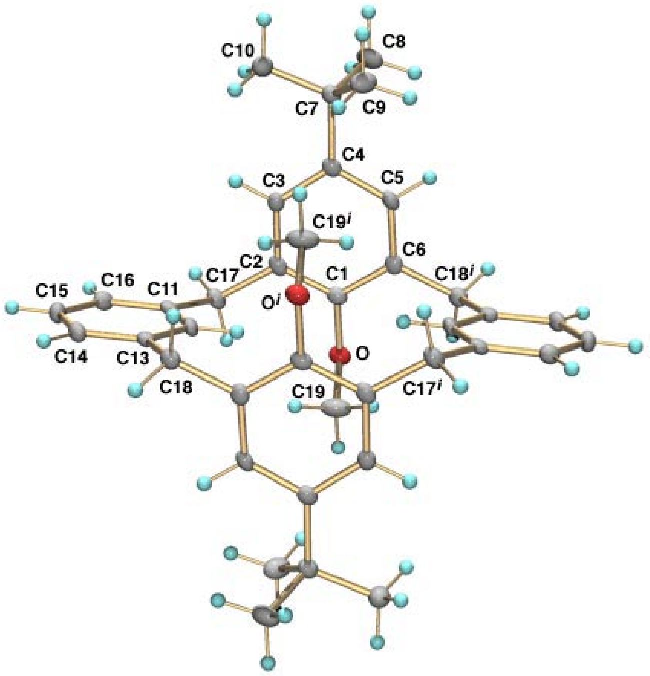


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids. Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Experimental

The title compound was prepared by alkylation of bis-9,12-*tert*-butyl-25,27-dihydrocalix[4]arene using methyl iodide and potassium carbonate in refluxing acetonitrile. Crystals suitable for X-ray diffraction grew from fractions obtained following chromatography (SiO_2 ether/hexanes eluent).

Crystal data

$C_{38}H_{44}O_2$
$M_r = 532.7$
Monoclinic, $P2_1/n$
$a = 8.6623$ (8) Å
$b = 18.1338$ (13) Å
$c = 9.8554$ (9) Å
$\beta = 105.68$ (8)°
$V = 1490.5$ (2) Å^3
$Z = 2$

$D_x = 1.187$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.1–14.3°$
$\mu = 0.07$ mm^{-1}
$T = 100$ K
Fragment, colourless
0.70 × 0.41 × 0.30 mm

Data collection

Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3130 measured reflections
2618 independent reflections
2090 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 10$
$k = -10 \rightarrow 21$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.03$
2618 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.4494P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O—C1	1.3891 (18)	O—C19	1.432 (2)
C1—O—C19	115.01 (14)	O—C1—C6	119.19 (15)
O—C1—C2	119.92 (15)		

A 1.1 mm collimator was used. All H atoms were placed in calculated positions, refined using a riding model, and given an isotropic displacement parameter equal to 1.2 (CH, CH_2) or 1.5 (CH_3) times the equivalent isotropic displacement parameter of the atom to which they were attached. The C—H distances used depended on the type of C atom: $C_{\rm methylene}$—H = 0.99, $C_{\rm methyl}$—H = 0.98, and $C_{\rm arene}$—H = 0.95 Å. Methyl-H atoms were allowed to rotate about the adjacent C—C bond.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993); cell refinement: *CAD*-4-*PC*; data reduction: *XCAD*-4 (Harms, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *PLATON* (Spek, 2001).

This research was sponsored by the Environmental Management Science Program of the Offices of Science and Environmental Management, US Department of Energy, under contract number DE–AC05–00OR22725 with Oak Ridge National Laboratory, managed and operated by UT–Battelle, LLC.

References

Bruker (1997). *SHELXTL*. Version 5.10/IRIX. Bruker AXS, Madison, Wisconsin, USA.
Enraf–Nonius (1993). *CAD*-4-*PC*. Version 1.2. Nonius, Delft, The Netherlands.
Grynszpan, F., Goren, Z. & Biali, S. E. (1991). *J. Org. Chem.* **56**, 532–536.
Harms, K. (1995). *XCAD*-4. Universität Marburg, Germany.
McMurry, J. E. & Phelan, J. C. (1991). *Tetrahedron Lett.* **32**, 5655–5658.
Perrin, M. & Oehler, D. (1991). *Calixarenes: A Versitile Class of Macrocyclic Compounds*, edited by J. Vicens and V. Böhmer, pp. 65–85. Dordrecht: Kluwer.
Sachleben, R. A., Urvoas, A., Bryan, J. C., Haverlock, T. J., Hay, B. P. & Moyer, B. A. (1999). *J. Chem. Soc. Chem. Commun.* pp. 1751–1752.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2001). *PLATON*. Utrecht University, The Netherlands.
Ting, Y., Verboom, W. & Reinhoudt, D. (1995). *Acta Cryst.* C**51**, 1465–1467.

Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

5,17-Di-*tert*-butyl-25,27-dihydroxycalix[4]arene. Erratum

Jeffrey C. Bryan,* Richard A. Sachleben and Agathe Urvoas

Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6119, USA

Correspondence e-mail: bryanjc@ornl.gov

In the paper by Bryan, Sachleben & Urvoas [*Acta Cryst.* E**57**, o781–o782], the chemical name in the title is incorrect. The correct title is 5,17-di-*tert*-butyl-25,27-dimethoxycalix[4]arene.

e10 Jeffrey C. Bryan *et al.* • $C_{38}H_{44}O_2$

DOI: 10.1107/S1600536801012934