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26,28-Bis(benzyloxy)-25,27-dihydroxy-5,17-dinitrocalix[4]arene methanol solvate

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In the title compound, $C_{42}H_{34}N_2O_8{\cdot}CH_4O$, the calix[4]arene assumes a cone conformation. The hydroxyl groups form intramolecular hydrogen bonds to the ether O atoms.

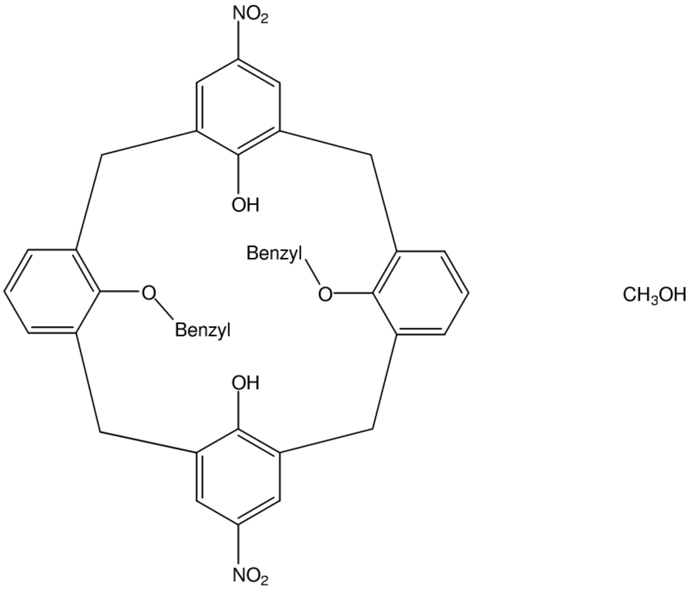
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
Some non-H atoms missing
Disorder in solvent or counterion
R factor = 0.057
wR factor = 0.153
Data-to-parameter ratio = 14.4

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

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).



(I)

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 (the average deviation from the mean plane is 0.147 Å). All four residues (the two hydroxyl 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 69.35 (7), 46.93 (7), 72.95 (6) and 38.16 (6)° for the rings C11–C16, C21–C26, C31–C36 and C41–C46, respectively. The torsion angles around the Ar—CH_2 bonds, which may be used to provide 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 (see Table 2 for details).

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Experimental

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

Crystal data

$C_{42}H_{34}N_2O_9 \cdot CH_4O$
$M_r = 726.75$
Monoclinic, $P2_1/n$
$a = 10.2000\ (8)$ Å
$b = 15.254\ (1)$ Å
$c = 22.986\ (2)$ Å
$\beta = 90.508\ (6)^\circ$
$V = 3576.3\ (5)$ Å^3
$Z = 4$

$D_x = 1.350$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 510 reflections
$\theta = 1$–20°
$\mu = 0.10$ mm^{-1}
$T = 173\ (2)$ K
Plate, yellow
0.35 × 0.32 × 0.11 mm

Data collection

Siemens SMART CCD three-circle diffractometer
ω scans
Absorption correction: none
67307 measured reflections
6793 independent reflections

4635 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.065$
$\theta_{\rm max} = 25.7^\circ$
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 18$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.153$
$S = 1.06$
6793 reflections
472 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 1.9514P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.45$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.47$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0021 (5)

Table 1
Selected geometric parameters (Å, °).

C12—O51	1.396 (3)	C42—O42	1.349 (3)
C22—O22	1.344 (3)	O51—C51	1.450 (3)
C32—O61	1.399 (3)	O61—C61	1.449 (3)
C12—O51—C51	112.71 (19)	C32—O61—C61	112.52 (17)
C43—C1—C11—C12	97.3 (3)	C23—C3—C31—C32	100.1 (3)
C21—C2—C13—C12	−105.2 (3)	C41—C4—C33—C32	−108.4 (3)
C13—C2—C21—C22	81.8 (3)	C33—C4—C41—C42	76.5 (3)
C31—C3—C23—C22	−78.9 (3)	C11—C1—C43—C42	−70.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O22—H22···O61	0.84	1.99	2.806 (2)	164
O42—H42···O51	0.84	1.92	2.703 (2)	155

All H atoms were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or 1.2 $U_{\rm eq}({\rm O})$] using a riding model, with O—H = 0.84 Å and C—H = 0.95 and 0.99 Å for aromatic and methylene C atoms, respectively. There is approximately one molecule of disordered methanol per asymmetric unit which has been suppressed using the SQUEEZE option (van der Sluis & Spek, 1990) in *PLATON* (Spek, 2003).

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

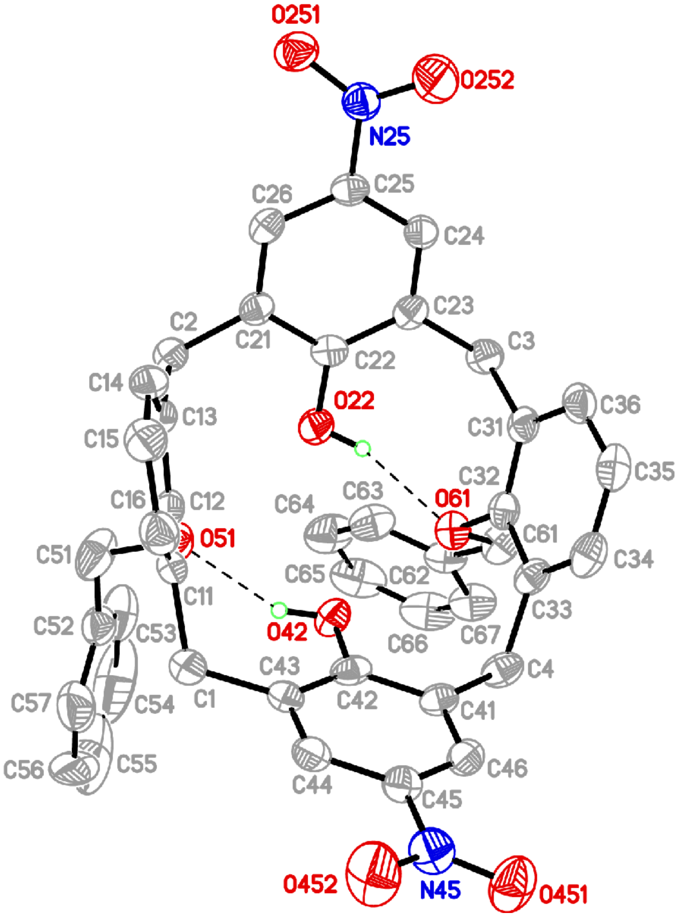

Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level and C-bound H atoms have been omitted for clarity. The MeOH nolecule is not shown. Hydrogen bonds are shown as dashed lines.

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