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25,27-Bis(acryloyloxy)-26,28-dihydroxycalix[4]arene toluene hemisolvate

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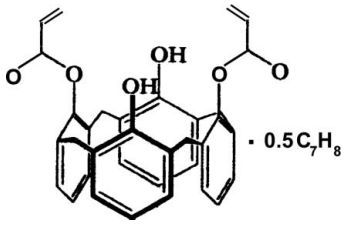
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.006 Å; R factor = 0.067; wR factor = 0.190; data-to-parameter ratio = 17.0.

In the title compound, $C_{34}H_{28}O_6\cdot0.5C_7H_8$, the calix[4]arene molecule forms a cone-like conformation, stabilized by intramolecular O—H···O hydrogen bonds. In the crystal structure, the calixarene molecules form centrosymmetric dimers *via* aromatic π–π interactions [centroid separation = 3.633 (3) Å]. The toluene solvent molecule is disordered about an inversion centre.

Related literature

For related literature, see: Andreetti *et al.* (1991); Böhmer (1995); Casnati *et al.* (1995); Gutsche (1989); Gutsche & Lin (1986); Kim *et al.* (1999, 2000); Vicens & Böhmer (1991).



Experimental

Crystal data

$C_{34}H_{28}O_6\cdot0.5C_7H_8$
M_r = 578.64
Triclinic, $P\overline{1}$
a = 10.5574 (9) Å
b = 12.0375 (12) Å
c = 13.5570 (12) Å
α = 111.105 (7)°
β = 101.460 (8)°
γ = 101.413 (7)°
V = 1505.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 297 (2) K
0.35 × 0.22 × 0.05 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.950, T_{max} = 0.993
23943 measured reflections
7061 independent reflections
3345 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.190
S = 0.98
7061 reflections
415 parameters
24 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H23A···O4	0.79 (8)	2.11 (8)	2.855 (4)	157 (8)
O3—H23B···O2	0.79 (8)	2.19 (7)	2.927 (4)	156 (7)
O1—H22A···O4	0.79 (7)	2.07 (7)	2.857 (4)	171 (6)
O1—H22B···O2	0.91 (11)	2.00 (11)	2.897 (3)	170 (9)

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2467).

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supplementary materials

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25,27-Bis(acryloyloxy)-26,28-dihydroxycalix[4]arene toluene hemisolvate

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Comment

Calixarenes are macrocyclic molecules made up of *p*-substituted phenolic units linked by methylene bridges *ortho* to the OH functions (Gutsche *et al.*, 1989; Vicens & Böhmer, 1991; Böhmer, 1995). The ester and ether derivatives of calix[4]arenes compounds can exist in one of the four conformations: cone, partial cone, 1,2-alternate, or 1,3-alternate (Andreetti *et al.*, 1991; Casnati *et al.*, 1995; Kim *et al.*, 1999; Kim *et al.*, 2000). As part of our work on calix[4]arene derivatives, we report herein the crystal structure of (I).

The asymmetric unit of (I) comprises one calixarene molecule and half of a toluene solvate molecule. The crystal structure of the 25,27-diacryloyloxy-26,28-dihydroxycalix[4]arene molecule is found in a cone conformation, stabilized by intramolecular hydrogen bonds involving the two phenol hydroxide groups and the two ester O atoms (Fig. 1). The mean plane defined by the four methylene C-atom bridges was chosen as a reference plane. The plane defined by the two phenol and the two ester O atoms makes a dihedral angle of 1.15 (12)° with this reference plane, whereas the dihedral angles between the reference plane and four aromatic rings are 37.61 (10)° for C1–C6 ring, 77.75 (8)° for C8–C13 ring, 33.55 (9)° for C15–C20 and 74.58 (9)° for C22–C27 ring. Cone conformation thus appears to be irregular and the aromatic rings containing the acryloyloxy groups are more inclined with respect to the reference plane than other rings. The bond angles involving the bridging methylene groups, *i.e.* C6—C7—C8 [111.89 (24)°] and C10—C14—C15 [113.42 (27)°] are significantly larger than the nominal tetrahedral angle due to repulsion among the four phenyl groups.

In the extended structure of (I) two calix[4]arene molecules are joined by strong π–π interactions (Fig. 2) between two phenyl (C1–C6, centroid = A) rings, which leads to the formation of a centrosymmetric dimer of (I) with an inter-planar separation of 3.484 Å for the A rings. The closest interatomic distance is C2···C6^{ii} [3.501 (5) Å]; symmetry code (ii): $1-x, 2-y, 1-z$] and the distance between the ring centroids is 3.633 (3) Å. These dimers are linked by van der Waals interactions.

Experimental

The title calixarene was synthesized according to the literature method of Gutsche & Lin (1986) and colourless plates of (I) were recrystallized from toluene.

Refinement

The C-bound H atoms were placed at calculated positions (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2$_{eq}$(C) or 1.5$_{eq}$(methyl C). The toluene molecule is disordered about an inversion centre. The O-bound H atoms were located in a difference map and their positions and U_{iso} values were freely refined. The hydrogen atoms of the phenol hydroxide groups, H23 and H22, are disordered over two positions, with occupancies of 0.52 and 0.48 for H23 and 0.6 and 0.4 for H22.

Figures

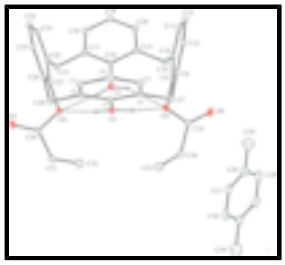


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level and C-bound H atoms are omitted for clarity (symmetry code i: −*x*, −*y*, 2 − *z*). H bonds are indicated by thin lines and both methyl disorder components of the toluene molecule are shown.

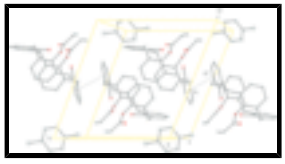


Fig. 2. Packing of (I), showing the π–π interactions between the symmetry related A rings [symmetry code (ii) 1 − *x*, 2 − *y*, 1 − *z*].

25,27-Bis(acryloyloxy)-26,28-dihydroxycalix[4]arene toluene hemisolvate

Crystal data

$C_{34}H_{28}O_6 \cdot 0.5C_7H_8$	$Z = 2$
$M_r = 578.64$	$F_{000} = 610.0$
Triclinic, $P\overline{1}$	$D_x = 1.277$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 10.5574$ (9) Å	Cell parameters from 4587 reflections
$b = 12.0375$ (12) Å	$\theta = 2.1$–28.1º
$c = 13.5570$ (12) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 111.105$ (7)º	$T = 297$ (2) K
$\beta = 101.460$ (8)º	Plate, colourless
$\gamma = 101.413$ (7)º	0.35 × 0.22 × 0.05 mm
$V = 1505.3$ (3) Å^3	

Data collection

Stoe IPDS II diffractometer	7061 independent reflections
Radiation source: fine-focus sealed tube	3345 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.076$
Detector resolution: 6.67 pixels mm^{-1}	$\theta_{max} = 27.8$º
$T = 297(2)$ K	$\theta_{min} = 2.2$º
ω scans	$h = -13 \rightarrow 13$
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$k = -15 \rightarrow 15$
$T_{min} = 0.950$, $T_{max} = 0.993$	$l = -17 \rightarrow 17$
23943 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.067$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.190$	$w = 1/[\sigma^2(F_o^2) + (0.088P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} < 0.001$
7061 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
415 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
24 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.008 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.4518 (3)	0.1542 (3)	0.5309 (2)	0.0459 (7)	
C2	0.5654 (3)	0.1796 (3)	0.4946 (3)	0.0464 (7)	
C3	0.5539 (3)	0.1091 (3)	0.3852 (3)	0.0564 (8)	
H3	0.6278	0.1238	0.3590	0.068*	
C4	0.4370 (4)	0.0182 (3)	0.3142 (3)	0.0583 (8)	
H4	0.4321	−0.0278	0.2411	0.070*	
C5	0.3269 (3)	−0.0045 (3)	0.3517 (3)	0.0553 (8)	
H5	0.2476	−0.0656	0.3030	0.066*	
C6	0.3319 (3)	0.0616 (3)	0.4602 (3)	0.0462 (7)	
C7	0.2101 (3)	0.0369 (3)	0.5012 (3)	0.0516 (7)	
H7A	0.1458	−0.0425	0.4488	0.062*	
H7B	0.2388	0.0313	0.5714	0.062*	
C8	0.1415 (3)	0.1384 (3)	0.5159 (3)	0.0494 (7)	
C9	0.1484 (3)	0.2252 (3)	0.6182 (3)	0.0482 (7)	
C10	0.1026 (3)	0.3289 (3)	0.6341 (3)	0.0538 (8)	

C11	0.0364 (3)	0.3383 (4)	0.5397 (3)	0.0631 (9)
H11	0.0024	0.4055	0.5466	0.076*
C12	0.0202 (3)	0.2503 (4)	0.4361 (3)	0.0663 (10)
H12	−0.0274	0.2568	0.3739	0.080*
C13	0.0737 (3)	0.1527 (3)	0.4236 (3)	0.0579 (8)
H13	0.0647	0.0953	0.3530	0.069*
C14	0.1282 (3)	0.4312 (3)	0.7471 (3)	0.0639 (9)
H14A	0.1582	0.4018	0.8030	0.077*
H14B	0.0439	0.4492	0.7537	0.077*
C15	0.2328 (3)	0.5503 (3)	0.7697 (3)	0.0561 (8)
C16	0.3706 (3)	0.5574 (3)	0.7870 (3)	0.0516 (7)
C17	0.4677 (3)	0.6665 (3)	0.8094 (3)	0.0530 (8)
C18	0.4256 (4)	0.7705 (3)	0.8167 (3)	0.0647 (9)
H18	0.4891	0.8448	0.8322	0.078*
C19	0.2910 (4)	0.7658 (4)	0.8015 (3)	0.0712 (10)
H19	0.2650	0.8370	0.8079	0.085*
C20	0.1961 (4)	0.6570 (3)	0.7770 (3)	0.0648 (9)
H20	0.1055	0.6542	0.7649	0.078*
C21	0.6166 (3)	0.6716 (3)	0.8272 (3)	0.0594 (8)
H21A	0.6711	0.7580	0.8583	0.071*
H21B	0.6423	0.6376	0.8804	0.071*
C22	0.6467 (3)	0.6001 (3)	0.7214 (3)	0.0519 (8)
C23	0.6462 (3)	0.6443 (3)	0.6394 (3)	0.0567 (8)
H23	0.6314	0.7211	0.6513	0.068*
C24	0.6672 (3)	0.5761 (3)	0.5408 (3)	0.0585 (8)
H24	0.6696	0.6086	0.4882	0.070*
C25	0.6846 (3)	0.4602 (3)	0.5200 (3)	0.0555 (8)
H25	0.6950	0.4139	0.4521	0.067*
C26	0.6868 (3)	0.4111 (3)	0.5993 (3)	0.0489 (7)
C27	0.6728 (3)	0.4861 (3)	0.6993 (3)	0.0495 (7)
C28	0.6952 (3)	0.2796 (3)	0.5727 (3)	0.0568 (8)
H28A	0.7177	0.2691	0.6410	0.068*
H28B	0.7679	0.2684	0.5396	0.068*
C30	0.7950 (3)	0.4717 (3)	0.8597 (3)	0.0574 (8)
C31	0.7816 (4)	0.4132 (4)	0.9374 (3)	0.0780 (11)
H31	0.8602	0.4225	0.9888	0.094*
C32	0.6698 (6)	0.3508 (6)	0.9391 (4)	0.121 (2)
H32A	0.5892	0.3396	0.8888	0.146*
H32B	0.6691	0.3165	0.9905	0.146*
C33	0.1407 (4)	0.1712 (3)	0.7707 (3)	0.0626 (9)
C34	0.2195 (5)	0.1432 (4)	0.8553 (4)	0.0880 (13)
H34	0.1797	0.1298	0.9070	0.106*
C35	0.3378 (6)	0.1362 (5)	0.8623 (4)	0.1059 (16)
H35A	0.3801	0.1492	0.8118	0.127*
H35B	0.3831	0.1181	0.9181	0.127*
C36	0.0956 (7)	0.0376 (7)	1.1018 (5)	0.122 (2)
H36	0.1598	0.0579	1.1683	0.146*
C37	0.0331 (7)	0.1154 (7)	1.0852 (5)	0.125 (2)
H37	0.0512	0.1954	1.1392	0.150*

C38	−0.0607 (6)	0.0733 (7)	0.9839 (6)	0.1193 (19)	
C39	−0.1297 (11)	0.1588 (10)	0.9654 (9)	0.215 (4)	
H39A	−0.0967	0.1881	0.9155	0.323*	
H39B	−0.1136	0.2282	1.0345	0.323*	
H39C	−0.2251	0.1172	0.9339	0.323*	
O1	0.4567 (3)	0.2189 (2)	0.6387 (2)	0.0562 (6)	
O2	0.2170 (2)	0.2112 (2)	0.71237 (17)	0.0566 (6)	
O3	0.4109 (3)	0.4560 (3)	0.7850 (2)	0.0663 (7)	
O4	0.6756 (2)	0.4377 (2)	0.78113 (18)	0.0577 (6)	
O5	0.8960 (2)	0.5393 (3)	0.8629 (2)	0.0801 (8)	
O6	0.0231 (3)	0.1615 (3)	0.7535 (2)	0.0841 (8)	
H23A	0.490 (8)	0.464 (7)	0.802 (6)	0.07 (2)*	0.52
H23B	0.343 (8)	0.401 (7)	0.762 (6)	0.05 (2)*	0.48
H22A	0.523 (7)	0.275 (6)	0.676 (5)	0.07 (2)*	0.60
H22B	0.377 (10)	0.207 (9)	0.654 (8)	0.08 (3)*	0.40

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0448 (15)	0.0475 (17)	0.0498 (18)	0.0174 (13)	0.0159 (13)	0.0222 (15)
C2	0.0461 (15)	0.0477 (17)	0.0567 (19)	0.0206 (13)	0.0214 (14)	0.0275 (15)
C3	0.0596 (19)	0.064 (2)	0.067 (2)	0.0308 (17)	0.0316 (17)	0.0382 (19)
C4	0.069 (2)	0.061 (2)	0.0515 (19)	0.0277 (18)	0.0234 (17)	0.0244 (17)
C5	0.0583 (19)	0.0524 (18)	0.0533 (19)	0.0181 (15)	0.0120 (15)	0.0215 (16)
C6	0.0456 (16)	0.0467 (17)	0.0520 (18)	0.0156 (13)	0.0147 (14)	0.0253 (15)
C7	0.0456 (16)	0.0473 (17)	0.0552 (19)	0.0053 (13)	0.0125 (14)	0.0197 (16)
C8	0.0363 (14)	0.0570 (18)	0.0542 (19)	0.0059 (13)	0.0132 (13)	0.0263 (16)
C9	0.0312 (13)	0.0604 (19)	0.0526 (19)	0.0069 (13)	0.0117 (13)	0.0269 (17)
C10	0.0353 (14)	0.061 (2)	0.067 (2)	0.0120 (14)	0.0216 (15)	0.0256 (18)
C11	0.0402 (16)	0.071 (2)	0.088 (3)	0.0216 (16)	0.0209 (17)	0.040 (2)
C12	0.0463 (18)	0.082 (3)	0.074 (3)	0.0173 (18)	0.0077 (17)	0.043 (2)
C13	0.0428 (16)	0.068 (2)	0.0545 (19)	0.0052 (15)	0.0093 (14)	0.0256 (17)
C14	0.0507 (18)	0.064 (2)	0.080 (2)	0.0179 (16)	0.0356 (18)	0.023 (2)
C15	0.0547 (18)	0.060 (2)	0.058 (2)	0.0205 (16)	0.0269 (16)	0.0222 (17)
C16	0.0495 (17)	0.0505 (18)	0.0569 (19)	0.0170 (14)	0.0216 (15)	0.0204 (16)
C17	0.0544 (18)	0.0522 (19)	0.0514 (18)	0.0116 (15)	0.0201 (15)	0.0203 (16)
C18	0.075 (2)	0.055 (2)	0.065 (2)	0.0125 (18)	0.0239 (18)	0.0285 (18)
C19	0.082 (3)	0.066 (2)	0.081 (3)	0.035 (2)	0.032 (2)	0.037 (2)
C20	0.059 (2)	0.073 (2)	0.074 (2)	0.0318 (18)	0.0272 (18)	0.033 (2)
C21	0.0508 (17)	0.058 (2)	0.057 (2)	0.0054 (15)	0.0140 (15)	0.0161 (17)
C22	0.0389 (15)	0.0515 (18)	0.0581 (19)	0.0023 (13)	0.0144 (14)	0.0205 (16)
C23	0.0482 (17)	0.0513 (18)	0.072 (2)	0.0089 (14)	0.0175 (16)	0.0304 (18)
C24	0.0544 (18)	0.063 (2)	0.065 (2)	0.0095 (16)	0.0212 (16)	0.0358 (19)
C25	0.0451 (16)	0.063 (2)	0.061 (2)	0.0107 (15)	0.0232 (15)	0.0278 (18)
C26	0.0327 (14)	0.0543 (18)	0.062 (2)	0.0096 (13)	0.0149 (13)	0.0276 (17)
C27	0.0326 (14)	0.0597 (19)	0.0529 (19)	0.0030 (13)	0.0107 (13)	0.0261 (16)
C28	0.0396 (15)	0.068 (2)	0.072 (2)	0.0195 (15)	0.0208 (15)	0.0342 (19)
C30	0.0387 (16)	0.073 (2)	0.0532 (19)	0.0180 (15)	0.0077 (14)	0.0199 (18)

C31	0.066 (2)	0.107 (3)	0.061 (2)	0.021 (2)	0.0057 (18)	0.043 (2)
C32	0.102 (4)	0.169 (6)	0.093 (4)	0.002 (4)	0.010 (3)	0.085 (4)
C33	0.063 (2)	0.064 (2)	0.052 (2)	0.0000 (17)	0.0156 (17)	0.0241 (18)
C34	0.083 (3)	0.106 (3)	0.070 (3)	0.008 (3)	0.022 (2)	0.043 (3)
C35	0.105 (4)	0.132 (4)	0.094 (4)	0.032 (3)	0.030 (3)	0.063 (3)
C36	0.098 (4)	0.144 (6)	0.086 (4)	−0.015 (4)	0.024 (3)	0.035 (4)
C37	0.101 (4)	0.146 (6)	0.093 (4)	−0.022 (4)	0.027 (3)	0.043 (4)
C38	0.090 (4)	0.163 (6)	0.116 (4)	0.015 (4)	0.049 (3)	0.073 (5)
C39	0.210 (9)	0.253 (11)	0.208 (9)	0.091 (8)	0.081 (7)	0.102 (9)
O1	0.0465 (13)	0.0603 (15)	0.0544 (15)	0.0086 (12)	0.0181 (11)	0.0178 (13)
O2	0.0466 (11)	0.0705 (15)	0.0520 (13)	0.0100 (10)	0.0145 (10)	0.0285 (12)
O3	0.0466 (14)	0.0524 (15)	0.101 (2)	0.0152 (12)	0.0252 (14)	0.0315 (15)
O4	0.0397 (11)	0.0768 (15)	0.0567 (13)	0.0072 (10)	0.0074 (10)	0.0365 (12)
O5	0.0409 (12)	0.104 (2)	0.0860 (18)	0.0099 (13)	0.0083 (12)	0.0405 (17)
O6	0.0602 (16)	0.105 (2)	0.0789 (18)	−0.0054 (14)	0.0260 (14)	0.0422 (16)

Geometric parameters (Å, °)

C1—O1	1.371 (4)	C21—H21B	0.9700
C1—C6	1.398 (4)	C22—C27	1.392 (4)
C1—C2	1.403 (4)	C22—C23	1.394 (4)
C2—C3	1.385 (5)	C23—C24	1.382 (5)
C2—C28	1.517 (4)	C23—H23	0.9300
C3—C4	1.372 (5)	C24—C25	1.378 (5)
C3—H3	0.9300	C24—H24	0.9300
C4—C5	1.375 (5)	C25—C26	1.400 (4)
C4—H4	0.9300	C25—H25	0.9300
C5—C6	1.382 (4)	C26—C27	1.388 (4)
C5—H5	0.9300	C26—C28	1.516 (5)
C6—C7	1.518 (4)	C27—O4	1.425 (3)
C7—C8	1.514 (4)	C28—H28A	0.9700
C7—H7A	0.9700	C28—H28B	0.9700
C7—H7B	0.9700	C30—O5	1.189 (4)
C8—C9	1.380 (4)	C30—O4	1.356 (4)
C8—C13	1.397 (4)	C30—C31	1.476 (5)
C9—C10	1.388 (4)	C31—C32	1.277 (6)
C9—O2	1.420 (4)	C31—H31	0.9300
C10—C11	1.385 (5)	C32—H32A	0.9300
C10—C14	1.510 (5)	C32—H32B	0.9300
C11—C12	1.373 (5)	C33—O6	1.191 (4)
C11—H11	0.9300	C33—O2	1.372 (4)
C12—C13	1.373 (5)	C33—C34	1.460 (6)
C12—H12	0.9300	C34—C35	1.256 (6)
C13—H13	0.9300	C34—H34	0.9300
C14—C15	1.514 (4)	C35—H35A	0.9300
C14—H14A	0.9700	C35—H35B	0.9300
C14—H14B	0.9700	C36—C37	1.304 (9)
C15—C20	1.389 (5)	C36—C38^{i}	1.337 (9)
C15—C16	1.406 (4)	C36—H36	0.9300

C16—O3	1.363 (4)	C37—C38	1.372 (9)
C16—C17	1.387 (4)	C37—H37	0.9300
C17—C18	1.387 (5)	C38—C36^{i}	1.337 (9)
C17—C21	1.526 (5)	C38—C39	1.437 (8)
C18—C19	1.381 (5)	C39—H39A	0.9600
C18—H18	0.9300	C39—H39B	0.9600
C19—C20	1.366 (5)	C39—H39C	0.9600
C19—H19	0.9300	O1—H22A	0.79 (7)
C20—H20	0.9300	O1—H22B	0.91 (11)
C21—C22	1.517 (5)	O3—H23A	0.79 (8)
C21—H21A	0.9700	O3—H23B	0.79 (8)
O1—C1—C6	117.8 (3)	C22—C21—H21B	109.0
O1—C1—C2	120.4 (3)	C17—C21—H21B	109.0
C6—C1—C2	121.8 (3)	H21A—C21—H21B	107.8
C3—C2—C1	117.2 (3)	C27—C22—C23	116.3 (3)
C3—C2—C28	121.6 (3)	C27—C22—C21	122.3 (3)
C1—C2—C28	121.2 (3)	C23—C22—C21	121.3 (3)
C4—C3—C2	122.0 (3)	C24—C23—C22	121.3 (3)
C4—C3—H3	119.0	C24—C23—H23	119.4
C2—C3—H3	119.0	C22—C23—H23	119.4
C3—C4—C5	119.7 (3)	C25—C24—C23	120.3 (3)
C3—C4—H4	120.2	C25—C24—H24	119.9
C5—C4—H4	120.2	C23—C24—H24	119.9
C4—C5—C6	121.3 (3)	C24—C25—C26	121.1 (3)
C4—C5—H5	119.3	C24—C25—H25	119.4
C6—C5—H5	119.3	C26—C25—H25	119.4
C5—C6—C1	118.0 (3)	C27—C26—C25	116.3 (3)
C5—C6—C7	121.3 (3)	C27—C26—C28	122.7 (3)
C1—C6—C7	120.7 (3)	C25—C26—C28	120.8 (3)
C8—C7—C6	111.8 (2)	C26—C27—C22	124.4 (3)
C8—C7—H7A	109.3	C26—C27—O4	117.1 (3)
C6—C7—H7A	109.3	C22—C27—O4	118.3 (3)
C8—C7—H7B	109.3	C26—C28—C2	113.5 (2)
C6—C7—H7B	109.3	C26—C28—H28A	108.9
H7A—C7—H7B	107.9	C2—C28—H28A	108.9
C9—C8—C13	116.6 (3)	C26—C28—H28B	108.9
C9—C8—C7	123.1 (3)	C2—C28—H28B	108.9
C13—C8—C7	120.2 (3)	H28A—C28—H28B	107.7
C8—C9—C10	124.1 (3)	O5—C30—O4	123.1 (3)
C8—C9—O2	116.9 (3)	O5—C30—C31	125.8 (3)
C10—C9—O2	118.8 (3)	O4—C30—C31	111.2 (3)
C11—C10—C9	116.6 (3)	C32—C31—C30	124.7 (4)
C11—C10—C14	120.5 (3)	C32—C31—H31	117.6
C9—C10—C14	122.9 (3)	C30—C31—H31	117.6
C12—C11—C10	121.2 (3)	C31—C32—H32A	120.0
C12—C11—H11	119.4	C31—C32—H32B	120.0
C10—C11—H11	119.4	H32A—C32—H32B	120.0
C13—C12—C11	120.5 (3)	O6—C33—O2	122.8 (3)

C13—C12—H12	119.7	O6—C33—C34	124.5 (3)
C11—C12—H12	119.7	O2—C33—C34	112.7 (3)
C12—C13—C8	120.8 (3)	C35—C34—C33	123.8 (4)
C12—C13—H13	119.6	C35—C34—H34	118.1
C8—C13—H13	119.6	C33—C34—H34	118.1
C10—C14—C15	113.4 (3)	C34—C35—H35A	120.0
C10—C14—H14A	108.9	C34—C35—H35B	120.0
C15—C14—H14A	108.9	H35A—C35—H35B	120.0
C10—C14—H14B	108.9	C37—C36—C38^{i}	115.0 (7)
C15—C14—H14B	108.9	C37—C36—H36	122.5
H14A—C14—H14B	107.7	C38^{i}—C36—H36	122.5
C20—C15—C16	117.9 (3)	C36—C37—C38	116.7 (8)
C20—C15—C14	121.0 (3)	C36—C37—H37	121.6
C16—C15—C14	121.1 (3)	C38—C37—H37	121.6
O3—C16—C17	118.8 (3)	C36^{i}—C38—C37	128.3 (7)
O3—C16—C15	119.5 (3)	C36^{i}—C38—C39	114.9 (8)
C17—C16—C15	121.7 (3)	C37—C38—C39	116.8 (9)
C16—C17—C18	117.9 (3)	C38—C39—H39A	109.5
C16—C17—C21	120.7 (3)	C38—C39—H39B	109.5
C18—C17—C21	121.4 (3)	H39A—C39—H39B	109.5
C19—C18—C17	121.2 (3)	C38—C39—H39C	109.5
C19—C18—H18	119.4	H39A—C39—H39C	109.5
C17—C18—H18	119.4	H39B—C39—H39C	109.5
C20—C19—C18	120.1 (3)	C1—O1—H22A	116 (4)
C20—C19—H19	119.9	C1—O1—H22B	116 (6)
C18—C19—H19	119.9	H22A—O1—H22B	125 (8)
C19—C20—C15	121.1 (3)	C33—O2—C9	117.9 (2)
C19—C20—H20	119.5	C16—O3—H23A	118 (5)
C15—C20—H20	119.5	C16—O3—H23B	105 (5)
C22—C21—C17	113.0 (3)	H23A—O3—H23B	137 (8)
C22—C21—H21A	109.0	C30—O4—C27	117.7 (2)
C17—C21—H21A	109.0		
O1—C1—C2—C3	178.8 (3)	C16—C17—C18—C19	−0.5 (5)
C6—C1—C2—C3	0.2 (4)	C21—C17—C18—C19	−179.4 (3)
O1—C1—C2—C28	−0.8 (4)	C17—C18—C19—C20	−1.1 (6)
C6—C1—C2—C28	−179.4 (3)	C18—C19—C20—C15	1.7 (6)
C1—C2—C3—C4	0.2 (4)	C16—C15—C20—C19	−0.8 (5)
C28—C2—C3—C4	179.8 (3)	C14—C15—C20—C19	177.5 (3)
C2—C3—C4—C5	0.1 (5)	C16—C17—C21—C22	71.8 (4)
C3—C4—C5—C6	−0.7 (5)	C18—C17—C21—C22	−109.2 (4)
C4—C5—C6—C1	1.0 (4)	C17—C21—C22—C27	−106.3 (3)
C4—C5—C6—C7	179.7 (3)	C17—C21—C22—C23	72.1 (4)
O1—C1—C6—C5	−179.4 (3)	C27—C22—C23—C24	1.6 (4)
C2—C1—C6—C5	−0.7 (4)	C21—C22—C23—C24	−176.8 (3)
O1—C1—C6—C7	1.9 (4)	C22—C23—C24—C25	2.3 (5)
C2—C1—C6—C7	−179.5 (3)	C23—C24—C25—C26	−2.6 (5)
C5—C6—C7—C8	−105.1 (3)	C24—C25—C26—C27	−1.0 (4)
C1—C6—C7—C8	73.6 (3)	C24—C25—C26—C28	175.3 (3)

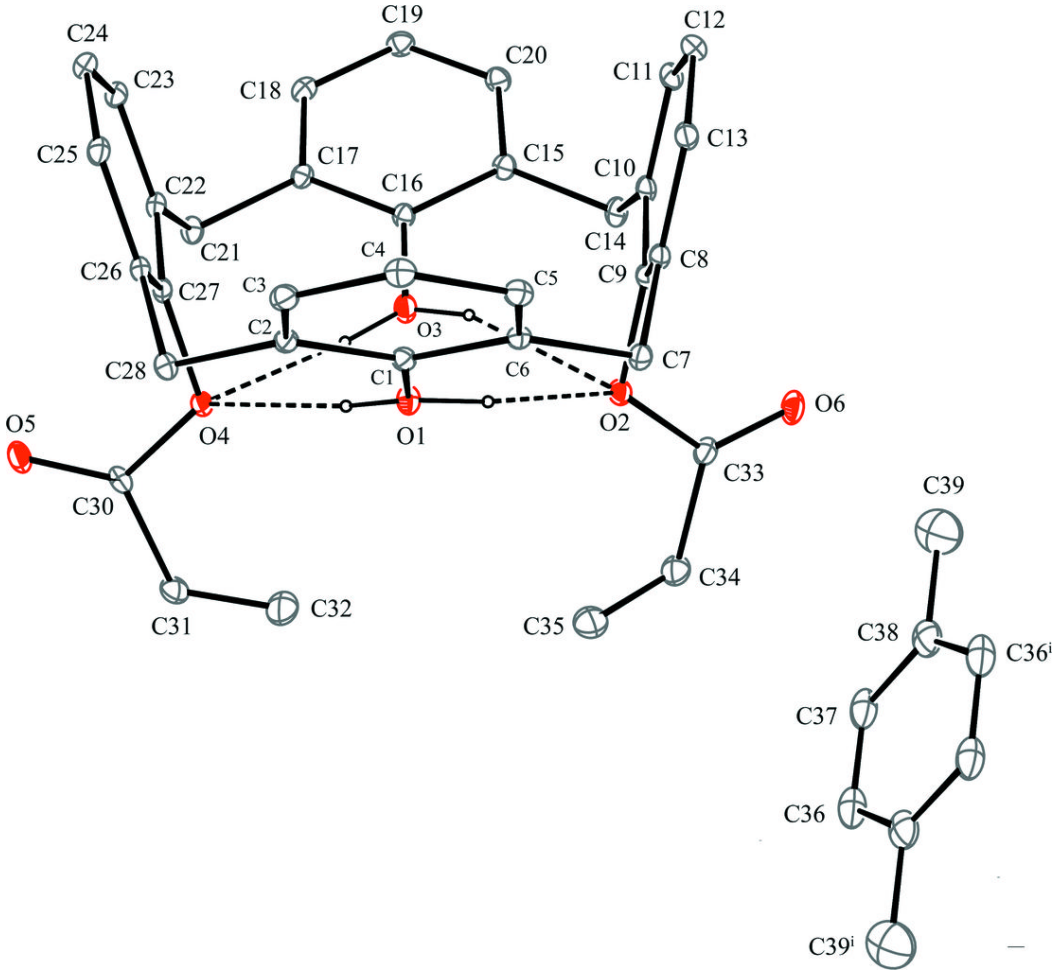
C6—C7—C8—C9	−107.8 (3)	C25—C26—C27—C22	5.2 (4)
C6—C7—C8—C13	68.3 (3)	C28—C26—C27—C22	−171.0 (3)
C13—C8—C9—C10	−5.4 (4)	C25—C26—C27—O4	−179.7 (2)
C7—C8—C9—C10	170.8 (3)	C28—C26—C27—O4	4.1 (4)
C13—C8—C9—O2	179.9 (2)	C23—C22—C27—C26	−5.5 (4)
C7—C8—C9—O2	−3.9 (4)	C21—C22—C27—C26	172.9 (3)
C8—C9—C10—C11	5.3 (4)	C23—C22—C27—O4	179.4 (2)
O2—C9—C10—C11	180.0 (2)	C21—C22—C27—O4	−2.2 (4)
C8—C9—C10—C14	−171.6 (3)	C27—C26—C28—C2	103.7 (3)
O2—C9—C10—C14	3.1 (4)	C25—C26—C28—C2	−72.3 (3)
C9—C10—C11—C12	−1.3 (4)	C3—C2—C28—C26	106.5 (3)
C14—C10—C11—C12	175.7 (3)	C1—C2—C28—C26	−73.9 (4)
C10—C11—C12—C13	−2.3 (5)	O5—C30—C31—C32	172.8 (5)
C11—C12—C13—C8	2.3 (5)	O4—C30—C31—C32	−7.7 (7)
C9—C8—C13—C12	1.4 (4)	O6—C33—C34—C35	169.2 (5)
C7—C8—C13—C12	−174.9 (3)	O2—C33—C34—C35	−11.2 (7)
C11—C10—C14—C15	−70.1 (4)	C38^{i}—C36—C37—C38	0.9 (9)
C9—C10—C14—C15	106.7 (3)	C36—C37—C38—C36^{i}	−1.1 (10)
C10—C14—C15—C20	110.7 (4)	C36—C37—C38—C39	180.0 (6)
C10—C14—C15—C16	−71.0 (4)	O6—C33—O2—C9	−8.7 (5)
C20—C15—C16—O3	177.3 (3)	C34—C33—O2—C9	171.6 (3)
C14—C15—C16—O3	−1.0 (5)	C8—C9—O2—C33	−108.0 (3)
C20—C15—C16—C17	−0.7 (5)	C10—C9—O2—C33	77.0 (3)
C14—C15—C16—C17	−179.1 (3)	O5—C30—O4—C27	0.0 (5)
O3—C16—C17—C18	−176.7 (3)	C31—C30—O4—C27	−179.5 (3)
C15—C16—C17—C18	1.3 (5)	C26—C27—O4—C30	96.2 (3)
O3—C16—C17—C21	2.2 (5)	C22—C27—O4—C30	−88.3 (3)
C15—C16—C17—C21	−179.7 (3)		

Symmetry codes: (i) −*x*, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H23A···O4	0.79 (8)	2.11 (8)	2.855 (4)	157 (8)
O3—H23B···O2	0.79 (8)	2.19 (7)	2.927 (4)	156 (7)
O1—H22A···O4	0.79 (7)	2.07 (7)	2.857 (4)	171 (6)
O1—H22B···O2	0.91 (11)	2.00 (11)	2.897 (3)	170 (9)

Fig. 1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C36
C39i

Fig. 2