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25,27-Bis(acryloyloxy)-5,11,17,23-tetra-*tert*-butyl-26,28-dihydroxycalix[4]arene

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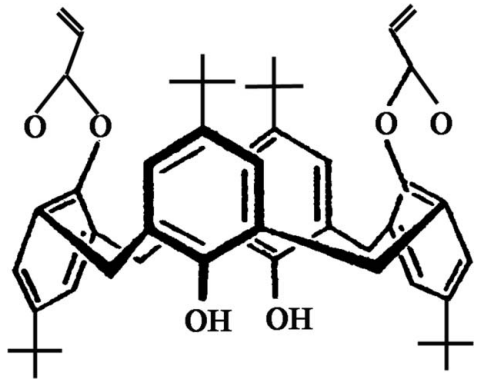
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.058; wR factor = 0.153; data-to-parameter ratio = 14.7.

The title compound, $C_{50}H_{60}O_6$, is a new *p-tert*-butylcalix[4]-arene derivative, adopting a 1,3-alternate conformation, with the complete molecule generated by twofold rotation symmetry. In the crystal structure, the molecules associate into layers in the *ac* plane. The three methyl groups of one *tert*-butyl group are disordered over two positions with site-occupancy factors approximately in the ratio 3:1.

Related literature

For related literature, see: Andreetti *et al.* (1991); Casnati *et al.* (1995); Gutsche (1989); Gutsche & Alam (1988); Gutsche & Lin (1986); Iwamoto *et al.* (1991); Kim *et al.* (1999, 2000).



Experimental

Crystal data

$C_{50}H_{60}O_6$
M_r = 756.98
Monoclinic, $C2/c$
a = 15.8896 (11) Å
b = 26.482 (2) Å
c = 10.3522 (7) Å
β = 95.047 (6)°
V = 4339.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 297 (2) K
0.41 × 0.24 × 0.09 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.940, T_{max} = 0.983
28360 measured reflections
4277 independent reflections
1985 reflections with $I > 2\sigma(I)$
R_{int} = 0.131

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.153
S = 0.89
4277 reflections
291 parameters
69 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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: HB2469).

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supplementary materials

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25,27-Bis(acryloyloxy)-5,11,17,23-tetra-*tert*-butyl-26,28-dihydroxycalix[4]arene

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Comment

For the past 20 years the calixarenes, cavity-shaped macrocycles, have attracted much attention mainly in supramolecular and analytical chemistry, because they can form typical host–guest complexes with many neutral molecules and ions, like cyclodextrins and crown ethers (Gutsche & Alam, 1988; Gutsche, 1989). Owing to their nonplanar structure, calix[4]arenes can exist in one of the four conformations, and has been designated as cone, partial cone, 1,2-alternate, and 1,3-alternate [Andreetti *et al.*, 1991; Casnati *et al.*, 1995; Kim *et al.*, 1999; Kim *et al.*, 2000). By placing substituents at OH groups larger than methyl, conformation can be locked. Very often cone and partial cone conformes were synthesized by the alkylation (Iwamoto *et al.*, 1991) and acylation (Gutsche & Lin, 1986) reaction at lover rim of calix[4]arene. But 1,2-alternate and 1,3-alternate conformers were observed only under certain reaction conditions.

As part of our work on substituted calix[4]arenes, we report herein the crystal structure of the title compound, (I), adopting a 1,3-alternate conformation. The two phenyl groups, A and D, lie above and the other two phenyl groups, B and C, below the least-squares plane defined by the four bridging methylene group, as illustrated in Fig. 1. The complete molecule is generated by 2-fold rotation symmetry. Bond angles involving the bridging methylene groups, i.e, C5—C7—C8 [116.2 (2)°] and C3—C18—C19 [116.4 (2)°], are significantly larger than the tetrahedral angle due to repulsion among the four phenyl groups. The dihedral angles of two pairs of facing rings, namely A and D, to which the OH group is bonded, and B and C, to which the acryloiloxy group is bonded, are 35.34 (10) and 20.01 (11)° respectively, so that rings A and D are splayed out upwards, and C and B are splayed out downwards from the central axis. Dihedral angles of adjacent phenyl rings in the calix[4]arene range from 85.13 (7) to 88.82 (7)°.

In the extended structure, there are no hydrogen bonding interactions and van der Waals interactions stabilize the extended structure (Fig. 2).

Experimental

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

Refinement

The H atoms were placed at calculated positions (O—H = 0.82 Å, C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2$_{eq}$(C) or 1.5U_{eq}(O, methyl C).

The disordered tetra-*tert*-butyl moiety [site-occupancy factors of 0.753 (9) for C44A/C88A/C99A and 0.247 (9) for C44B/C88B/C99B] was refined anisotropically, with constraints and restraints imposed on the anisotropic displacement parameters of C atoms.

Figures

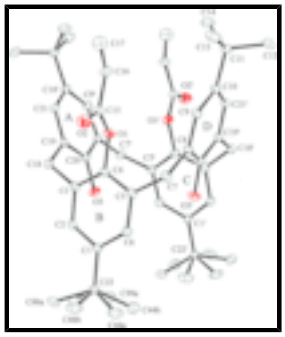

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level and H atoms are omitted for the clarity. Symmetry code: (i) 1 − *x*, *y*, 0.5 − *z*.

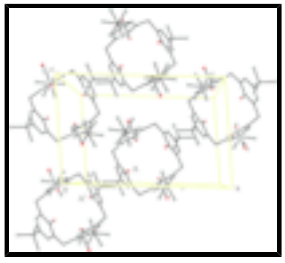

Fig. 2. The extended structure of (I).

25,27-Bis(acryloyloxy)-5,11,17,23-tetra-*tert*-butyl-26,28-dihydroxycalix[4]arene

Crystal data

$C_{50}H_{60}O_6$	F_{000} = 1632
M_r = 756.98	D_x = 1.158 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71069 Å
Hall symbol: -C 2yc	Cell parameters from 2487 reflections
a = 15.8896 (11) Å	θ = 2.0–28.1º
b = 26.482 (2) Å	μ = 0.07 mm^{-1}
c = 10.3522 (7) Å	*T* = 297 (2) K
β = 95.047 (6)º	Plate, colorless
V = 4339.2 (5) Å^3	0.41 × 0.24 × 0.09 mm
Z = 4	

Data collection

Stoe IPDS2 diffractometer	4277 independent reflections
Radiation source: fine-focus sealed tube	1985 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.131
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 26.0º
T = 297(2) K	θ_{min} = 2.4º
ω scans	*h* = −19→19
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	*k* = −32→32

T_{min} = 0.940, T_{max} = 0.983
28360 measured reflections

l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.153
S = 0.89
4277 reflections
291 parameters
69 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0017 (3)

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	0.39759 (18)	0.14689 (10)	0.4886 (3)	0.0489 (7)	
C2	0.33804 (18)	0.17650 (10)	0.4175 (3)	0.0516 (7)	
H2	0.2892	0.1612	0.3802	0.062*	
C3	0.34844 (17)	0.22828 (10)	0.3998 (3)	0.0502 (7)	
C4	0.42153 (18)	0.24978 (10)	0.4597 (2)	0.0488 (7)	
C5	0.48335 (17)	0.22247 (10)	0.5324 (2)	0.0484 (7)	
C6	0.46870 (18)	0.17095 (11)	0.5453 (3)	0.0511 (7)	
H6	0.5087	0.1518	0.5945	0.061*	
C7	0.56389 (18)	0.24571 (11)	0.5930 (3)	0.0556 (7)	
H7A	0.5994	0.2189	0.6311	0.067*	
H7B	0.5499	0.2676	0.6630	0.067*	
C8	0.61498 (17)	0.27597 (10)	0.5029 (2)	0.0477 (7)	
C9	0.63760 (17)	0.32534 (10)	0.5299 (3)	0.0522 (7)	
H9	0.6215	0.3399	0.6057	0.063*	

C10	0.68349 (18)	0.35436 (10)	0.4487 (3)	0.0522 (7)	
C11	0.7106 (2)	0.40896 (11)	0.4792 (3)	0.0611 (8)	
C12	0.8071 (2)	0.41237 (13)	0.4860 (4)	0.0823 (11)	
H12A	0.8245	0.4463	0.5076	0.123*	
H12B	0.8259	0.4034	0.4034	0.123*	
H12C	0.8313	0.3896	0.5512	0.123*	
C13	0.6815 (3)	0.42761 (13)	0.6066 (4)	0.0915 (12)	
H13A	0.6209	0.4265	0.6025	0.137*	
H13B	0.7004	0.4617	0.6218	0.137*	
H13C	0.7047	0.4064	0.6760	0.137*	
C14	0.6726 (2)	0.44401 (12)	0.3718 (4)	0.0833 (11)	
H14A	0.6121	0.4433	0.3700	0.125*	
H14B	0.6894	0.4328	0.2897	0.125*	
H14C	0.6924	0.4778	0.3884	0.125*	
C15	0.41891 (19)	0.33598 (11)	0.5224 (3)	0.0613 (8)	
C16	0.4375 (2)	0.38666 (13)	0.4723 (5)	0.0894 (12)	
H16	0.4549	0.3896	0.3891	0.107*	
C17	0.4307 (3)	0.4257 (2)	0.5384 (7)	0.153 (2)	
H17A	0.4134	0.4234	0.6218	0.183*	
H17B	0.4429	0.4571	0.5043	0.183*	
C18	0.28391 (17)	0.25805 (11)	0.3157 (3)	0.0549 (7)	
H18A	0.2628	0.2849	0.3679	0.066*	
H18B	0.2367	0.2359	0.2900	0.066*	
C19	0.31394 (16)	0.28140 (10)	0.1945 (2)	0.0477 (7)	
C20	0.35920 (17)	0.25403 (10)	0.1088 (3)	0.0475 (6)	
C21	0.29447 (17)	0.33092 (10)	0.1629 (3)	0.0509 (7)	
H21	0.2648	0.3496	0.2200	0.061*	
C22	0.38507 (18)	0.08963 (11)	0.4972 (3)	0.0563 (7)	
O1	0.43527 (12)	0.30121 (7)	0.43395 (18)	0.0581 (5)	
O2	0.39125 (18)	0.32601 (9)	0.6231 (3)	0.0931 (8)	
O3	0.37741 (13)	0.20369 (7)	0.1318 (2)	0.0658 (6)	
H3	0.4029	0.1924	0.0726	0.099*	
C88A	0.2920 (3)	0.07769 (17)	0.5133 (7)	0.088 (2)	0.753 (9)
H88A	0.2857	0.0421	0.5275	0.133*	0.753 (9)
H88B	0.2579	0.0875	0.4362	0.133*	0.753 (9)
H88C	0.2743	0.0960	0.5862	0.133*	0.753 (9)
C99A	0.4087 (6)	0.0663 (2)	0.3724 (6)	0.101 (3)	0.753 (9)
H99A	0.4678	0.0715	0.3644	0.152*	0.753 (9)
H99B	0.3763	0.0818	0.3004	0.152*	0.753 (9)
H99C	0.3969	0.0308	0.3729	0.152*	0.753 (9)
C44A	0.4365 (4)	0.06675 (19)	0.6111 (6)	0.099 (3)	0.753 (9)
H44A	0.4234	0.0315	0.6167	0.148*	0.753 (9)
H44B	0.4235	0.0834	0.6893	0.148*	0.753 (9)
H44C	0.4955	0.0707	0.6003	0.148*	0.753 (9)
C44B	0.4719 (9)	0.0619 (5)	0.505 (2)	0.095 (7)	0.247 (9)
H44D	0.5050	0.0718	0.5832	0.142*	0.247 (9)
H44E	0.5014	0.0708	0.4314	0.142*	0.247 (9)
H44F	0.4628	0.0261	0.5061	0.142*	0.247 (9)
C88B	0.3461 (16)	0.0783 (6)	0.6233 (19)	0.105 (8)	0.247 (9)

H88D	0.3407	0.0424	0.6332	0.157*	0.247 (9)
H88E	0.2913	0.0937	0.6213	0.157*	0.247 (9)
H88F	0.3818	0.0916	0.6950	0.157*	0.247 (9)
C99B	0.3324 (15)	0.0678 (6)	0.3805 (18)	0.102 (7)	0.247 (9)
H99D	0.3377	0.0317	0.3809	0.153*	0.247 (9)
H99E	0.3522	0.0810	0.3023	0.153*	0.247 (9)
H99F	0.2743	0.0769	0.3846	0.153*	0.247 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0487 (16)	0.0494 (17)	0.0488 (15)	−0.0049 (13)	0.0054 (13)	0.0044 (13)
C2	0.0460 (16)	0.0515 (17)	0.0573 (17)	−0.0068 (13)	0.0054 (14)	0.0032 (13)
C3	0.0496 (16)	0.0519 (17)	0.0507 (16)	−0.0022 (13)	0.0127 (14)	0.0065 (13)
C4	0.0568 (17)	0.0441 (16)	0.0476 (15)	−0.0075 (13)	0.0160 (14)	0.0049 (13)
C5	0.0516 (17)	0.0543 (18)	0.0401 (14)	−0.0081 (13)	0.0078 (13)	0.0046 (13)
C6	0.0524 (17)	0.0537 (18)	0.0468 (15)	−0.0035 (13)	0.0011 (14)	0.0086 (13)
C7	0.0596 (18)	0.0583 (18)	0.0482 (16)	−0.0138 (15)	0.0006 (14)	0.0008 (13)
C8	0.0477 (16)	0.0484 (17)	0.0461 (15)	−0.0067 (13)	−0.0016 (13)	0.0013 (13)
C9	0.0520 (17)	0.0531 (18)	0.0508 (16)	−0.0078 (13)	0.0014 (14)	−0.0068 (13)
C10	0.0538 (17)	0.0466 (16)	0.0563 (17)	−0.0097 (13)	0.0051 (14)	−0.0070 (13)
C11	0.070 (2)	0.0463 (17)	0.0679 (19)	−0.0130 (14)	0.0108 (17)	−0.0066 (14)
C12	0.076 (2)	0.057 (2)	0.113 (3)	−0.0241 (17)	0.004 (2)	−0.0104 (19)
C13	0.120 (3)	0.068 (2)	0.089 (3)	−0.032 (2)	0.027 (2)	−0.0326 (19)
C14	0.103 (3)	0.047 (2)	0.099 (3)	−0.0034 (18)	0.002 (2)	−0.0021 (18)
C15	0.0581 (19)	0.0485 (19)	0.078 (2)	−0.0042 (14)	0.0078 (17)	0.0011 (16)
C16	0.076 (2)	0.046 (2)	0.144 (4)	0.0032 (17)	−0.003 (2)	−0.018 (2)
C17	0.116 (4)	0.098 (4)	0.248 (7)	−0.001 (3)	0.037 (4)	−0.009 (4)
C18	0.0484 (17)	0.0525 (17)	0.0646 (18)	0.0048 (13)	0.0103 (15)	0.0075 (14)
C19	0.0418 (15)	0.0505 (17)	0.0508 (16)	0.0034 (12)	0.0050 (13)	0.0027 (13)
C20	0.0485 (15)	0.0397 (15)	0.0537 (16)	0.0064 (12)	0.0014 (13)	0.0001 (12)
C21	0.0534 (17)	0.0448 (17)	0.0551 (16)	0.0116 (12)	0.0081 (14)	−0.0003 (13)
C22	0.0590 (18)	0.0488 (17)	0.0605 (17)	−0.0030 (13)	0.0028 (15)	0.0060 (14)
O1	0.0704 (13)	0.0460 (12)	0.0593 (12)	−0.0106 (9)	0.0126 (10)	0.0055 (9)
O2	0.122 (2)	0.0752 (16)	0.0875 (18)	0.0008 (14)	0.0381 (17)	−0.0087 (13)
O3	0.0848 (15)	0.0450 (12)	0.0699 (13)	0.0151 (10)	0.0190 (12)	0.0017 (10)
C88A	0.081 (3)	0.050 (3)	0.137 (6)	−0.020 (2)	0.025 (4)	0.008 (3)
C99A	0.154 (7)	0.065 (3)	0.088 (4)	0.014 (4)	0.036 (4)	−0.009 (3)
C44A	0.117 (6)	0.064 (3)	0.107 (5)	−0.016 (3)	−0.032 (4)	0.026 (3)
C44B	0.079 (8)	0.053 (9)	0.15 (2)	0.003 (7)	0.007 (8)	−0.029 (10)
C88B	0.15 (2)	0.060 (10)	0.112 (11)	0.009 (10)	0.069 (14)	0.018 (9)
C99B	0.127 (16)	0.049 (8)	0.121 (11)	−0.015 (10)	−0.039 (12)	−0.016 (9)

Geometric parameters (Å, °)

C1—C6	1.382 (4)	C16—H16	0.9300
C1—C2	1.389 (4)	C17—H17A	0.9300
C1—C22	1.533 (4)	C17—H17B	0.9300
C2—C3	1.395 (4)	C18—C19	1.513 (4)

C2—H2	0.9300	C18—H18A	0.9700
C3—C4	1.389 (4)	C18—H18B	0.9700
C3—C18	1.508 (4)	C19—C21	1.380 (4)
C4—C5	1.388 (4)	C19—C20	1.394 (3)
C4—O1	1.409 (3)	C20—O3	1.380 (3)
C5—C6	1.393 (4)	C20—C8^{i}	1.388 (4)
C5—C7	1.506 (4)	C21—C10^{i}	1.383 (4)
C6—H6	0.9300	C21—H21	0.9300
C7—C8	1.518 (4)	C22—C44A	1.502 (5)
C7—H7A	0.9700	C22—C99A	1.509 (5)
C7—H7B	0.9700	C22—C99B	1.522 (13)
C8—C9	1.378 (4)	C22—C88B	1.523 (12)
C8—C20^{i}	1.388 (4)	C22—C88A	1.535 (5)
C9—C10	1.392 (4)	C22—C44B	1.558 (12)
C9—H9	0.9300	O3—H3	0.8200
C10—C21^{i}	1.383 (4)	C88A—H88A	0.9600
C10—C11	1.534 (4)	C88A—H88B	0.9600
C11—C13	1.518 (4)	C88A—H88C	0.9600
C11—C14	1.531 (5)	C99A—H99A	0.9600
C11—C12	1.531 (4)	C99A—H99B	0.9600
C12—H12A	0.9600	C99A—H99C	0.9600
C12—H12B	0.9600	C44A—H44A	0.9600
C12—H12C	0.9600	C44A—H44B	0.9600
C13—H13A	0.9600	C44A—H44C	0.9600
C13—H13B	0.9600	C44B—H44D	0.9600
C13—H13C	0.9600	C44B—H44E	0.9600
C14—H14A	0.9600	C44B—H44F	0.9600
C14—H14B	0.9600	C88B—H88D	0.9600
C14—H14C	0.9600	C88B—H88E	0.9600
C15—O2	1.195 (3)	C88B—H88F	0.9600
C15—O1	1.340 (4)	C99B—H99D	0.9600
C15—C16	1.478 (5)	C99B—H99E	0.9600
C16—C17	1.250 (6)	C99B—H99F	0.9600
C6—C1—C2	117.3 (3)	C3—C18—C19	116.4 (2)
C6—C1—C22	122.5 (3)	C3—C18—H18A	108.2
C2—C1—C22	120.2 (3)	C19—C18—H18A	108.2
C1—C2—C3	122.8 (3)	C3—C18—H18B	108.2
C1—C2—H2	118.6	C19—C18—H18B	108.2
C3—C2—H2	118.6	H18A—C18—H18B	107.3
C4—C3—C2	116.6 (3)	C21—C19—C20	117.4 (2)
C4—C3—C18	122.9 (2)	C21—C19—C18	120.3 (2)
C2—C3—C18	120.5 (3)	C20—C19—C18	122.2 (2)
C5—C4—C3	123.6 (2)	O3—C20—C8^{i}	118.4 (2)
C5—C4—O1	119.5 (2)	O3—C20—C19	120.3 (2)
C3—C4—O1	116.7 (2)	C8^{i}—C20—C19	121.3 (2)
C4—C5—C6	116.5 (3)	C19—C21—C10^{i}	123.8 (2)
C4—C5—C7	123.2 (3)	C19—C21—H21	118.1

C6—C5—C7	120.2 (3)	$C10^{i}$—C21—H21	118.1
C1—C6—C5	123.2 (3)	C44A—C22—C99A	110.4 (4)
C1—C6—H6	118.4	C44A—C22—C99B	133.9 (7)
C5—C6—H6	118.4	C99A—C22—C99B	47.6 (9)
C5—C7—C8	116.2 (2)	C44A—C22—C88B	58.7 (9)
C5—C7—H7A	108.2	C99A—C22—C88B	143.5 (7)
C8—C7—H7A	108.2	C99B—C22—C88B	111.7 (13)
C5—C7—H7B	108.2	C44A—C22—C1	112.3 (3)
C8—C7—H7B	108.2	C99A—C22—C1	108.2 (3)
H7A—C7—H7B	107.4	C99B—C22—C1	113.3 (7)
$C9—C8—C20^{i}$	118.4 (2)	C88B—C22—C1	108.0 (6)
C9—C8—C7	121.4 (2)	C44A—C22—C88A	107.1 (4)
$C20^{i}$—C8—C7	120.3 (2)	C99A—C22—C88A	108.8 (4)
C8—C9—C10	122.9 (3)	C99B—C22—C88A	62.7 (10)
C8—C9—H9	118.6	C88B—C22—C88A	53.0 (10)
C10—C9—H9	118.6	C1—C22—C88A	109.9 (3)
$C21^{i}$—C10—C9	116.2 (2)	C44A—C22—C44B	49.5 (8)
$C21^{i}$—C10—C11	120.5 (2)	C99A—C22—C44B	64.4 (8)
C9—C10—C11	123.3 (2)	C99B—C22—C44B	106.6 (11)
C13—C11—C14	107.6 (3)	C88B—C22—C44B	106.6 (12)
C13—C11—C12	108.7 (3)	C1—C22—C44B	110.5 (6)
C14—C11—C12	109.1 (3)	C88A—C22—C44B	138.9 (7)
C13—C11—C10	112.6 (2)	C15—O1—C4	119.5 (2)
C14—C11—C10	109.7 (3)	C20—O3—H3	109.5
C12—C11—C10	109.1 (2)	C22—C88A—H88A	109.5
C11—C12—H12A	109.5	C22—C88A—H88B	109.5
C11—C12—H12B	109.5	C22—C88A—H88C	109.5
H12A—C12—H12B	109.5	C22—C99A—H99A	109.5
C11—C12—H12C	109.5	C22—C99A—H99B	109.5
H12A—C12—H12C	109.5	C22—C99A—H99C	109.5
H12B—C12—H12C	109.5	C22—C44A—H44A	109.5
C11—C13—H13A	109.5	C22—C44A—H44B	109.5
C11—C13—H13B	109.5	C22—C44A—H44C	109.5
H13A—C13—H13B	109.5	C22—C44B—H44D	109.5
C11—C13—H13C	109.5	C22—C44B—H44E	109.5
H13A—C13—H13C	109.5	H44D—C44B—H44E	109.5
H13B—C13—H13C	109.5	C22—C44B—H44F	109.5
C11—C14—H14A	109.5	H44D—C44B—H44F	109.5
C11—C14—H14B	109.5	H44E—C44B—H44F	109.5
H14A—C14—H14B	109.5	C22—C88B—H88D	109.5
C11—C14—H14C	109.5	C22—C88B—H88E	109.5
H14A—C14—H14C	109.5	H88D—C88B—H88E	109.5
H14B—C14—H14C	109.5	C22—C88B—H88F	109.5
O2—C15—O1	123.5 (3)	H88D—C88B—H88F	109.5
O2—C15—C16	127.3 (3)	H88E—C88B—H88F	109.5
O1—C15—C16	109.1 (3)	C22—C99B—H99D	109.5
C17—C16—C15	122.0 (5)	C22—C99B—H99E	109.5
C17—C16—H16	119.0	H99D—C99B—H99E	109.5

C15—C16—H16	119.0	C22—C99B—H99F	109.5
C16—C17—H17A	120.0	H99D—C99B—H99F	109.5
C16—C17—H17B	120.0	H99E—C99B—H99F	109.5
H17A—C17—H17B	120.0		
C6—C1—C2—C3	1.4 (4)	C9—C10—C11—C12	120.5 (3)
C22—C1—C2—C3	−176.0 (2)	O2—C15—C16—C17	5.0 (6)
C1—C2—C3—C4	−1.4 (4)	O1—C15—C16—C17	−177.2 (4)
C1—C2—C3—C18	176.7 (2)	C4—C3—C18—C19	62.2 (3)
C2—C3—C4—C5	1.1 (4)	C2—C3—C18—C19	−115.7 (3)
C18—C3—C4—C5	−176.9 (2)	C3—C18—C19—C21	−133.9 (3)
C2—C3—C4—O1	175.4 (2)	C3—C18—C19—C20	48.3 (4)
C18—C3—C4—O1	−2.6 (3)	C21—C19—C20—O3	−177.1 (3)
C3—C4—C5—C6	−0.8 (4)	C18—C19—C20—O3	0.8 (4)
O1—C4—C5—C6	−174.9 (2)	C21—C19—C20—C8^{i}	1.1 (4)
C3—C4—C5—C7	177.7 (2)	C18—C19—C20—C8^{i}	179.0 (3)
O1—C4—C5—C7	3.5 (4)	C20—C19—C21—C10^{i}	0.7 (4)
C2—C1—C6—C5	−1.1 (4)	C18—C19—C21—C10^{i}	−177.2 (3)
C22—C1—C6—C5	176.3 (2)	C6—C1—C22—C44A	22.8 (5)
C4—C5—C6—C1	0.8 (4)	C2—C1—C22—C44A	−159.9 (4)
C7—C5—C6—C1	−177.7 (2)	C6—C1—C22—C99A	−99.3 (5)
C4—C5—C7—C8	−52.3 (4)	C2—C1—C22—C99A	78.0 (5)
C6—C5—C7—C8	126.1 (3)	C6—C1—C22—C99B	−150.1 (12)
C5—C7—C8—C9	125.0 (3)	C2—C1—C22—C99B	27.2 (12)
C5—C7—C8—C20^{i}	−55.7 (4)	C6—C1—C22—C88B	85.7 (12)
C20^{i}—C8—C9—C10	1.5 (4)	C2—C1—C22—C88B	−97.0 (12)
C7—C8—C9—C10	−179.2 (3)	C6—C1—C22—C88A	142.0 (4)
C8—C9—C10—C21^{i}	0.2 (4)	C2—C1—C22—C88A	−40.7 (4)
C8—C9—C10—C11	−178.7 (3)	C6—C1—C22—C44B	−30.6 (10)
C21^{i}—C10—C11—C13	−179.1 (3)	C2—C1—C22—C44B	146.7 (10)
C9—C10—C11—C13	−0.3 (4)	O2—C15—O1—C4	−1.5 (5)
C21^{i}—C10—C11—C14	61.0 (4)	C16—C15—O1—C4	−179.5 (3)
C9—C10—C11—C14	−120.1 (3)	C5—C4—O1—C15	−83.6 (3)
C21^{i}—C10—C11—C12	−58.4 (4)	C3—C4—O1—C15	101.9 (3)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2.

Fig. 1

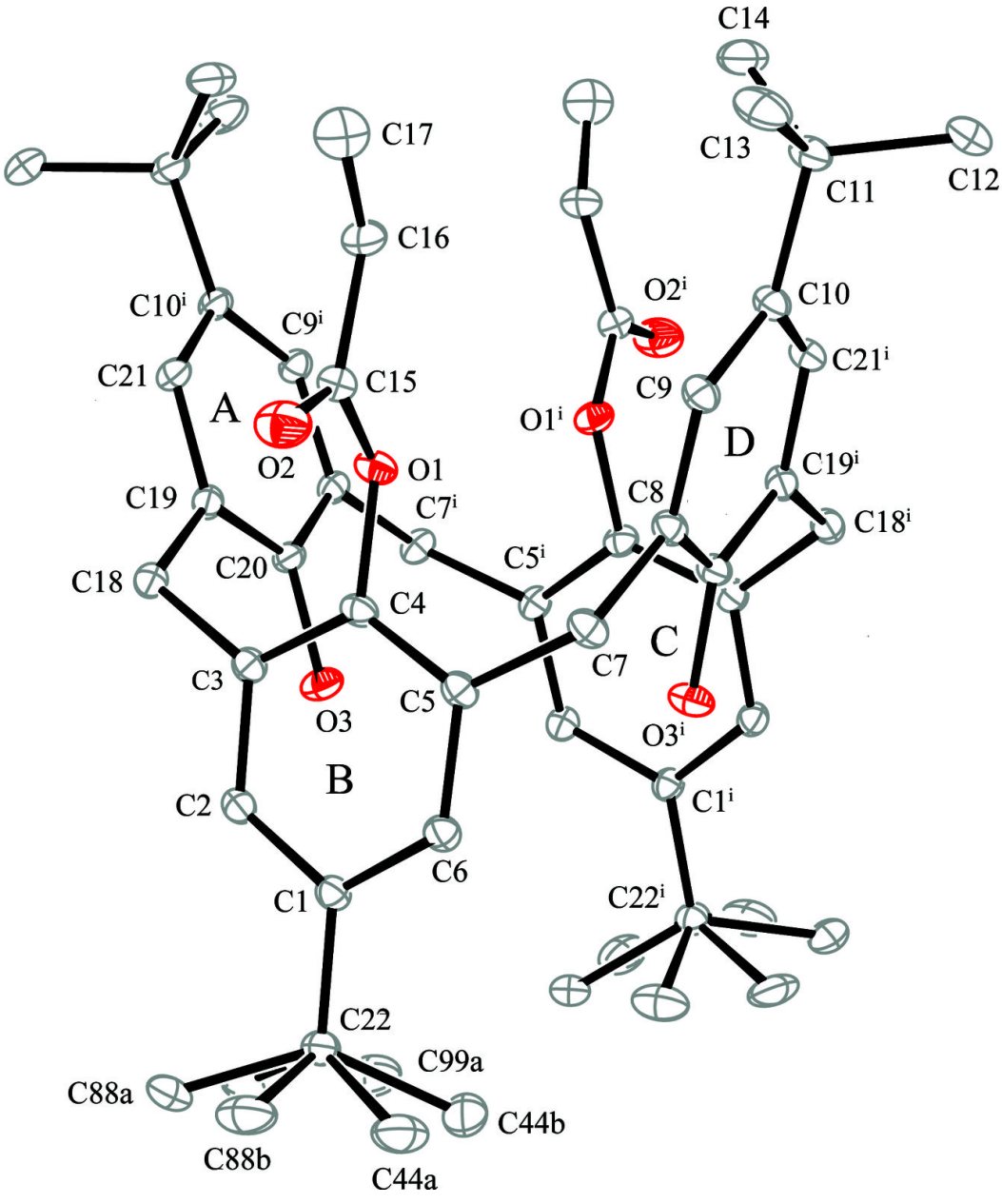
C14
C17
C13
C11
C12
C16
C10i
C9i
O2i
C10
C21
C15
C9
C21i
A
O2
O1i
D
O1
C19
C7i
C8
C19i
C18i
C18
C20
C5i
C4
C7
C
C3
O3
C5
O3i
B
C1i
C2
C6
C1
C22i
C22
C99a
C88a
C44b
C88b
C44a

Fig. 2