Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,7,12-Tris(4-bromobutoxy)-3,8,13-trimethoxy-10,15-dihydro-5*H*-tribenzo[*a*,*d*,*g*]cyclononene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.058
wR factor = 0.163
Data-to-parameter ratio = 16.9

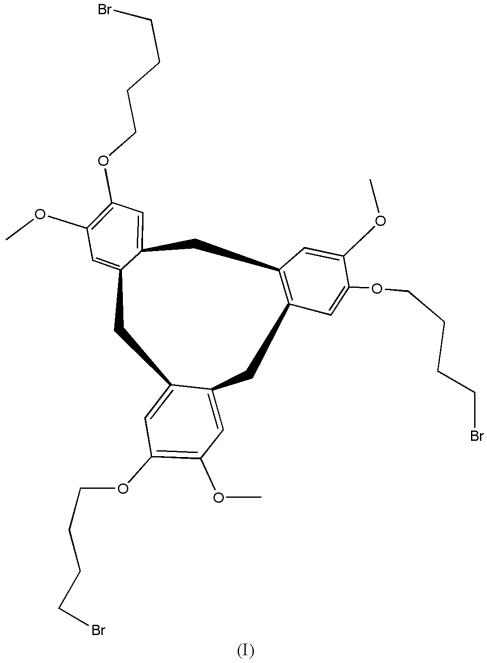
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 April 2004
Accepted 2 June 2004
Online 19 June 2004

The title compound, $C_{36}H_{45}O_6Br_3$, adopts the rigid 'crown' conformation of the parent cyclotriveratrylene (CTV) system and possesses molecular, but not crystallographic, C_3 symmetry. The cyclotriveratrylene core is rigid.

Comment

Cyclotriveratrylenes are excellent rigid bowl-shaped frameworks which have attracted current interest in the field of host–guest chemistry. In particular, the C_3 symmetry is attractive for the design of ligands capable of transition metal coordination, leading to complexes in which the metal could be accommodated in the cyclotriveratrylene cavity or placed in close proximity to this cavity (Thomas & Iyenbar, 1998). Furthermore, it can be used in the field of supramolecular chemistry. In this paper, the structure of 2,7,12-tris(4-bromobutoxy)-3,8,13-trimethoxy-10,15-dihydro-5*H*-tribenzo[*a*,*d*,*g*]-cyclononene, (I), is reported (Fig. 1).



(I)

The molecule adopts the rigid 'bowl' conformation of the parent cyclotriveratrylene (CTV) system (MacNicol, 1984) and possesses molecular, but not crystallographic, C_3 symmetry. Cyclotriveratrylene and several of its derivatives form crystal structures which are not closely packed and which can thus accommodate guest molecules within voids in their structure. This property is not necessarily associated with a crown conformation. On the other hand, the existence of a

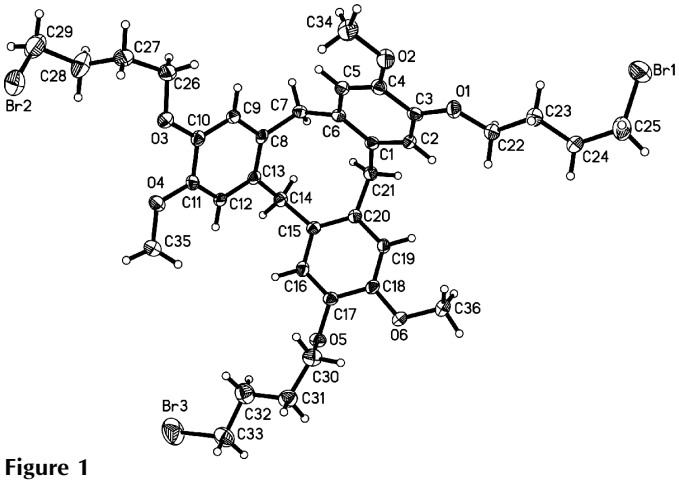


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. The minor occupancy disordered atoms have been omitted.

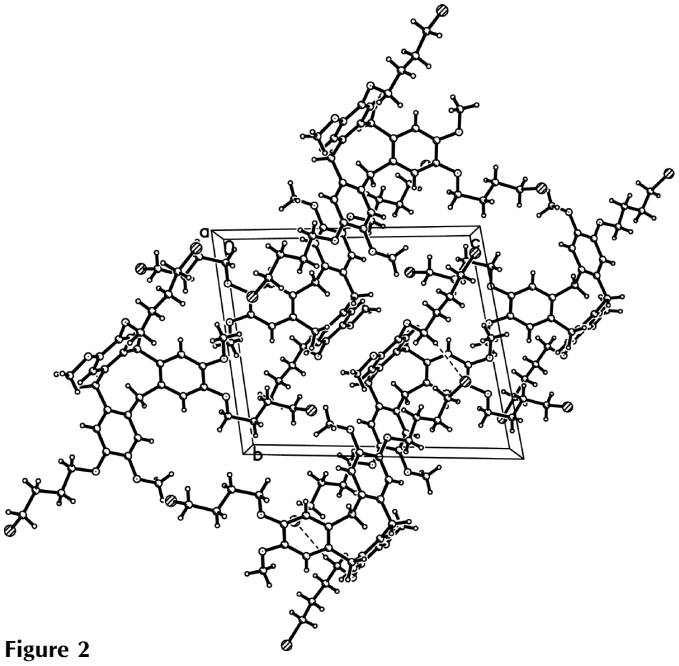


Figure 2
The crystal structure of (I), viewed along the *b* axis. Dashed lines indicate hydrogen.

rigid, bowl-shaped geometry is of great importance for the formation of host–guest molecular complexes (Collet, 1987).

Experimental

To a solution of [4-(2-bromoethoxy)-3-methoxyphenyl]methanol (5 g, 0.019 mol) in methanol (25 ml) was added 70% perchloric acid (25 g, 0.25 mol) dropwise with chilling and stirring. The mixture was stirred overnight at room temperature. Dilution with excess water and filtration gave a crude white solid. The solid was then chromatographed on a silica-gel column, eluting with ethyl acetate/petroleum ether (1/5), to afford the pure product (1.1 g, 23.6%). Colorless crystals were obtained by recrystallization from ethyl acetate.

Crystal data

$C_{36}H_{45}Br_3O_6$	$Z = 2$
$M_r = 813.45$	$D_x = 1.511$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.328$ (3) Å	Cell parameters from 1024 reflections
$b = 13.393$ (3) Å	$\theta = 3.7$–24.4°
$c = 15.694$ (4) Å	$\mu = 3.43$ mm^{-1}
$\alpha = 76.951$ (4)°	$T = 293$ (2) K
$\beta = 75.162$ (4)°	Block, colorless
$\gamma = 72.894$ (4)°	0.24 × 0.20 × 0.16 mm
$V = 1787.4$ (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	7248 independent reflections
φ and ω scans	3984 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$R_{int} = 0.027$
$T_{min} = 0.432$, $T_{max} = 0.578$	$\theta_{max} = 26.4^\circ$
10 436 measured reflections	$h = -11 \rightarrow 7$
	$k = -16 \rightarrow 12$
	$l = -18 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 1.5971P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.058$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.163$	$\Delta\rho_{max} = 0.85$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.92$ e Å^{-3}
7248 reflections	
428 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Atoms C33, Br3 and the H atoms on C32 and C33 are disordered, with occupancies of 0.807 (7) for H32*A*, H32*B*, C33, H33*A*, H33*B* and Br3, and 0.195 (7) for H33*C*, H33*D*, C33′, H33*C*, H33*D* and Br3′. All H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding, with U_{iso}= 1.2 or 1.5(methyl) U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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