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

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Key indicators

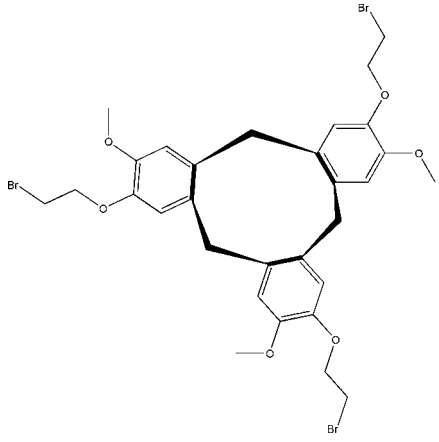
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.063
wR factor = 0.164
Data-to-parameter ratio = 16.0

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

The title compound, $C_{30}H_{33}Br_3O_6$, adopts a locked 'crown' conformation of the parent cyclotriveratrylene (CTV) system and possesses approximate C_3 symmetry. The three methoxy groups are coplanar with the benzene rings to which they are bound.

Comment

Cyclotriveratrylene and several of its derivatives form crystal structures which are not closely packed and which can thus accommodate guest molecules within voids. This property is not necessarily associated with a crown conformation. On the other hand, the existence of a rigid bowl-shaped geometry is of great importance for the formation of host–guest molecular complexes (Collet, 1987).



(I)

In this paper, the structure of 2,7,12-tris(2-bromoethoxy)-3,8,13-trimethoxy-10,15-dihydro-5*H*-tribenzo[*a*,*d*,*g*]cyclononaene, (I), is reported (Fig. 1). The molecule adopts a locked 'crown' conformation of the parent cyclotriveratrylene (CTV) system (MacNicol, 1984), and possesses essentially C_3 symmetry, a pseudo-threefold axis passing through the center of the crown, which is a nine-membered ring. The three methoxy groups of the crown are coplanar with the benzene rings to which they are bound.

Experimental

The title compound was synthesized as follows: to a solution of [4-(2-bromoethoxy)-3-methoxyphenyl]methanol (10 g, 0.038 mol) in methanol (54 ml) was added 70% perchloric acid (54.9 g, 0.55 mol) dropwise with chilling and stirring. The mixture was stirred overnight at room temperature. Dilution with excess water and filtration yielded a crude solid which was chromatographed on a silica-gel column, eluting with ethyl acetate/petroleum ether (1/5) to afford the pure product (2 g, 21.5%). Colorless crystals were obtained by recrystallization from ethyl acetate.

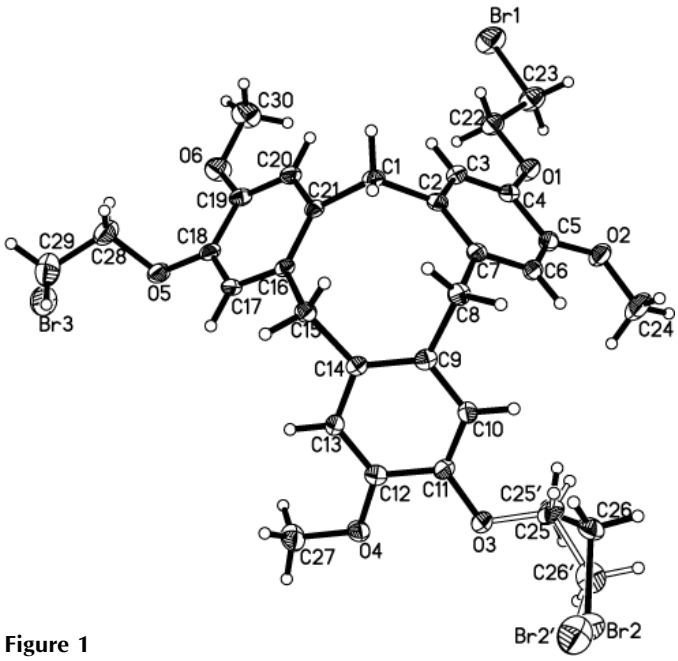


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. Both disorder components are shown.

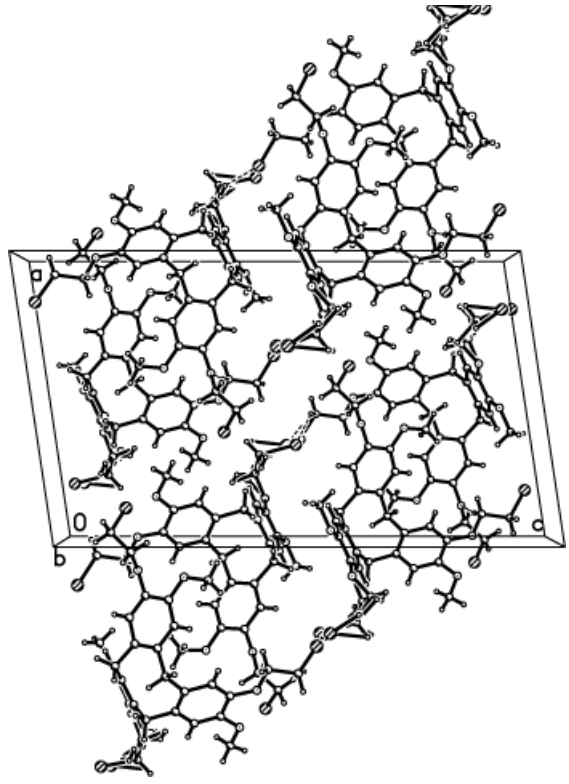


Figure 2
The crystal structure of (I), viewed along the *b* axis.

Crystal data

$C_{30}H_{33}Br_3O_6$	D_x = 1.609 Mg m^{-3}
M_r = 729.29	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 882 reflections
a = 14.585 (3) Å	θ = 3.1–23.7°
b = 8.3893 (19) Å	μ = 4.06 mm^{-1}
c = 24.881 (6) Å	T = 293 (2) K
β = 98.507 (4)°	Block, colorless
V = 3010.8 (12) Å^3	0.38 × 0.32 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	6129 independent reflections
φ and ω scans	3693 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.062
T_{min} = 0.263, T_{max} = 0.666	θ_{max} = 26.4°
18616 measured reflections	$h = -17 \rightarrow 18$
	$k = -9 \rightarrow 10$
	$l = -31 \rightarrow 30$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.164	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.01	$\Delta\rho_{max}$ = 0.69 e Å^{-3}
6129 reflections	$\Delta\rho_{min}$ = −0.85 e Å^{-3}
383 parameters	

Disorder was encountered in one of the 2-bromoethoxy chains. A twofold positional disorder model for this fragment was used to describe the situation and refined to occupancies 0.696 (11) and 0.304 (11) for the two sites. H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier). Disorder exists in one of the 2-bromo-ethoxy chains. There are two positions which are disordered. The bond lengths were restrained to 1.94 for C—Br, 1.54 for C—C and 1.44 Å for C—O.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); 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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