

A new pseudopolymorph of 5,11,17,23,- 29,35,41,47-octabromo-49,50,51,52,53,- 54,55,56-octamethoxycalix[8]arene

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$

R factor = 0.043

wR factor = 0.111

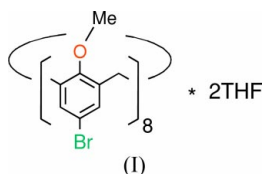
Data-to-parameter ratio = 16.2

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

Crystals of the title calixarene containing CCl_4 as solvent have already been investigated [Baudry, Felix, Bavoux, Perrin, Vocanson, Dumazet-Bonnamour & Lamartine (2003). *New J. Chem.* pp. 1540–1543]. We present here a new pseudopolymorph of this compound, $\text{C}_{64}\text{H}_{56}\text{Br}_8\text{O}_8 \cdot 2\text{C}_4\text{H}_8\text{O}$, containing tetrahydrofuran as solvent and crystallizing in a different space group. However, the molecular conformation of both molecules is very similar. Further similarities of both structures are that the calixarene molecules are located on a centre of inversion and that the solvent molecules occupy similar positions with respect to the calixarene molecules.

Comment

Recently, Baudry *et al.* (2003) published the crystal structure of 5,11,17,23,29,35,41,47-octabromo-49,50,51,52,53,54,55,56-octamethoxycalix[8]arene containing CCl_4 as solvent in the monoclinic space group $C2/c$, named (Ib) hereafter. We have obtained crystals of the same compound with tetrahydrofuran as solvent, (I). Interestingly, the space group is still monoclinic, but no longer centred, *viz.* $P2_1/n$. However, the molecular conformation of (I) does not change significantly. A least-squares fit of both structures (fitting the eight methylene C atoms) is shown in Fig. 2. A comparison of equivalent torsion angles is given in Table 1. A further similarity of both structures is that the calixarene molecules are located on a centre of inversion with just half a molecule in the asymmetric unit. Even the solvent molecules are located in similar positions with respect to the calixarene molecules as can be seen from Figs. 3 and 4.



Experimental

Compound (I) was prepared by *O*-methylation of *p*-bromocalix[8]arene with methyl iodide in tetrahydrofuran (Böhmer *et al.*, 2002). Single crystals were obtained by slow evaporation of a solution in tetrahydrofuran.

Crystal data

$\text{C}_{64}\text{H}_{56}\text{Br}_8\text{O}_8 \cdot 2\text{C}_4\text{H}_8\text{O}$

$M_r = 1736.58$

Monoclinic, $P2_1/n$

$a = 9.0636\text{ (7)\ \AA}$

$b = 26.2897\text{ (15)\ \AA}$

$c = 15.1205\text{ (11)\ \AA}$

$\beta = 93.287\text{ (6)^\circ}$

$V = 3597.0\text{ (4)\ \AA}^3$

$Z = 2$

$D_x = 1.603\text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

Cell parameters from 30 238

reflections

$\theta = 1.9\text{--}25.3^\circ$

$\mu = 4.52\text{ mm}^{-1}$

$T = 293\text{ (2)\ K}$

Block, colourless

$0.57 \times 0.54 \times 0.52\text{ mm}$

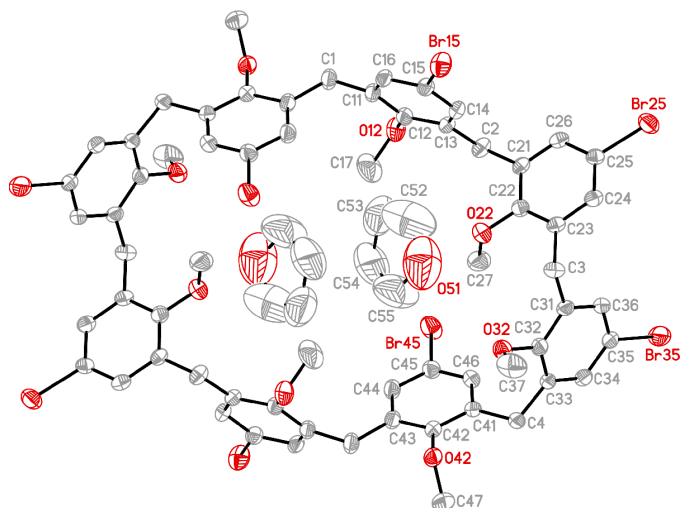


Figure 1
 Perspective view of (I), with the atom-numbering scheme (only the symmetry-independent atoms are labelled). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

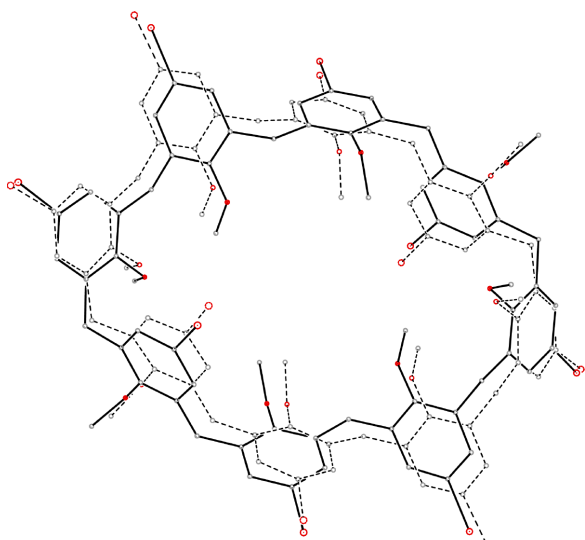


Figure 2
 Least-squares fit of (I) (dotted lines) and (Ib) (full lines).

Data collection

Stoe IPDS-II two-circle
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (MULABS; Spek, 1990; Blessing,
 1995)
 $T_{\min} = 0.086$, $T_{\max} = 0.094$
 56 958 measured reflections

6572 independent reflections
 4601 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.109$
 $\theta_{\text{max}} = 25.4^\circ$
 $h = -10 \rightarrow 10$
 $k = -31 \rightarrow 31$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.111$
 $S = 0.96$
 6572 reflections
 406 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.47 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.60 \text{ e } \text{\AA}^{-3}$

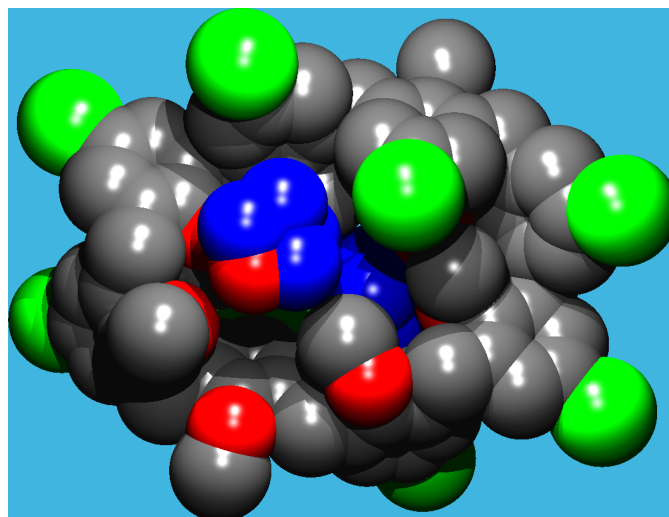


Figure 3
 CPK representation of (I), showing the position of the THF molecules with respect to the calixarene molecule. H atoms have been omitted. Colour code: C (except the THF C atoms) grey, Br green, Cl yellow, and O red. The C atoms of the THF molecules are drawn in blue.

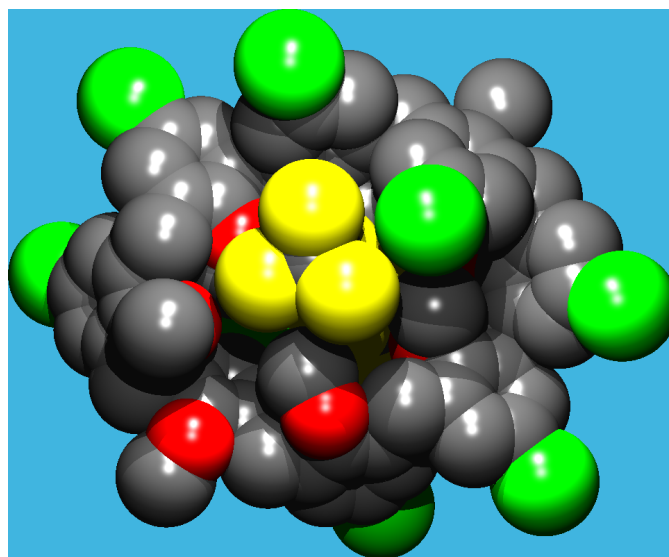


Figure 4
 CPK representation of (Ib), showing the position of the CCl_4 molecule with respect to the calixarene molecule; only the main conformation of the disordered Cl atoms is shown. H atoms have been omitted. Colour code: C grey, Br green, Cl yellow, and O red.

Table 1

Comparison of the torsion angles ($^\circ$) of (I) and (Ib).

	Compound (I)	Compound (Ib)
C42–C41–C4–C33	–146.0 (4)	–144.3
C41–C4–C33–C32	73.1 (5)	72.6
C32–C31–C3–C23	–134.6 (4)	–136.2
C31–C3–C23–C22	83.3 (5)	92.1
C22–C21–C2–C13	86.2 (5)	95.5
C21–C2–C13–C12	–164.3 (4)	–174.9
C12–C11–C1–C43 ⁱ	110.3 (5)	92.9
C11–C1–C43 ⁱ –C42 ⁱ	–175.2 (4)	–165.3

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

H atoms were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$] using a riding model, with aromatic C–H = 0.93 Å, methyl C–H = 0.96 Å and methylene C–H = 0.97 Å. The distances in the tetrahydrofuran molecule were restrained to 1.40 (1) and 1.55 (1) Å for C–O and C–C, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 1990).

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