Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis(cucurbit[6]uril) bis(hexane-1,6-diyldipyridinium) tetrabromide tridecahydrate

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Received 6 September 2006
Accepted 13 December 2006

Key indicators

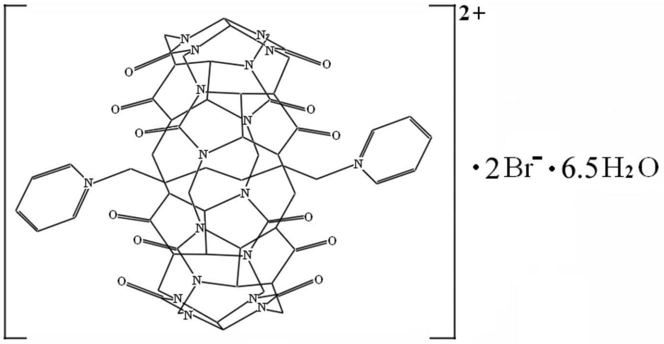
Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.033
wR factor = 0.085
Data-to-parameter ratio = 16.4

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

In the crystal structure of the title compound, $2C_{36}H_{36}N_{24}O_{12}\cdot 2C_{16}H_{22}N_2^{2+}\cdot 4Br^-\cdot 13H_2O$, the host cucurbituril is threaded by the guest hexane-1,6-diyldipyridinium cation to form a pseudorotaxane. Two Br^- anions form hydrogen bonds with two solvent water molecules, while the other solvent water molecules interact with the carbonyl O atoms at the rims of the cucurbituril.

Comment

The structure of the cucurbituril (Q[6]) molecular host (Freeman *et al.*, 1981) was first determined by single-crystal X-ray diffraction analysis in 1981. A series of cucurbituril homologues, analogues and derivatives has been reported in recent years (Day *et al.*, 2000, 2002; Kim *et al.*, 2000; Jon *et al.*, 2003; Lagona *et al.*, 2003). The guest-binding ability is exemplified by the inclusion of a series of long-chain organic compounds (Meschke *et al.*, 1999) and aromatic compounds (Buschmann & Wolff, 1999). In the present study, the title host–guest inclusion complex, (I), is presented. The complex consists of the host cucurbituril, $C_{36}H_{36}N_{24}O_{12}$, Q[6], a 1,6-hexylenedipyridinium guest cation, $C_{16}H_{22}N_2^{2+}$, and two bromide anions and 6.5 solvent water molecules. The host Q[6] is threaded by the guest cation and the bound site is the alkyl chain of the guest, forming a typical pseudorotaxane. The pyridinium rings at both ends are located outside the portals of the host Q[6] (Fig. 1).



(I)

In this host–guest inclusion complex, the molecular assembly has a plane of symmetry with two half-molecules related by the crystallographic symmetry operator (1 − *x*, −*y*, 1 − *z*). The hexyl chain is bound vertically inside the centre of the cavity of Q[6], with the six C atoms of the chain all enclosed, while the two pyridinium rings extend from the two portals (Fig. 2). Three water molecules interact with the O atoms of the carbonyl rim around each portal of the host

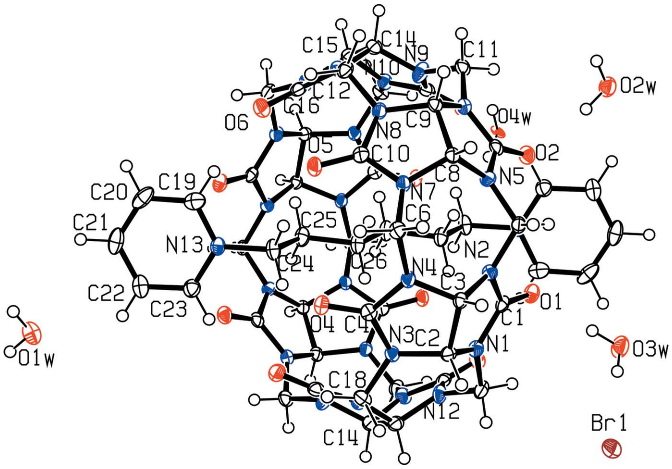


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

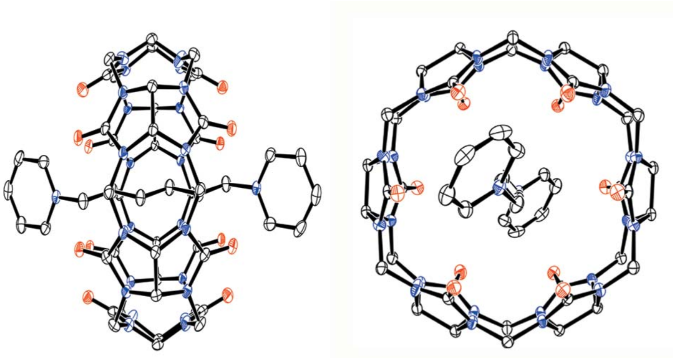


Figure 2
Plots of the structure of the host–guest complex, (I), of cucurbituril with 1,6-hexylenedipyridinium dibromide (50% probability displacement ellipsoids). On the left is a top view and on the right is a side view. Solvent water molecules, H atoms and bromide anions have been omitted for clarity.

through hydrogen-bond interactions, and two solvent water molecules also interact with the Br^- anions.

Experimental

The guest 1,6-hexylenedipyridinium dibromide (0.12 g, 0.30 mmol) was first dissolved in H_2O (80 ml), and to this solution cucurbit[6]uril (0.23 g, 0.20 mmol) was added. The mixture was heated to dissolve the host and guest and then filtered. The filtrate was set aside for one week to allow colourless crystals of (I) to deposit.

Crystal data

$2C_{36}H_{36}N_{24}O_{12}\cdot 2C_{16}H_{22}N_2^{2+}\cdot 4Br^-\cdot 13H_2O$
$M_r = 3032.34$
Monoclinic, $P2_1/c$
$a = 12.7444$ (6) Å
$b = 20.2909$ (8) Å
$c = 12.1950$ (5) Å
$\beta = 106.732$ (2)°
$V = 3020.1$ (2) Å^3
$Z = 1$
$D_x = 1.667$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.44$ mm^{-1}
$T = 123$ (2) K
Prism, colourless
$0.20 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.762$, $T_{max} = 0.813$
32085 measured reflections
7483 independent reflections
6334 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 28.3$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.085$
$S = 1.03$
7483 reflections
457 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 1.9327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.220 (2)	C3—N2	1.456 (2)
C1—N1	1.371 (2)	C4—O4	1.213 (2)
C1—N2	1.373 (2)	C4—N4	1.371 (2)
C2—N1	1.444 (2)	C4—N3	1.373 (2)
C2—N3	1.450 (2)	C5—N2	1.446 (2)
C2—C3	1.554 (2)	C6—N4	1.439 (2)
C3—N4	1.440 (2)		
O1—C1—N1	126.36 (16)	N4—C3—N2	114.84 (13)
O1—C1—N2	125.66 (15)	N4—C3—C2	103.13 (13)
N1—C1—N2	107.97 (14)	N2—C3—C2	103.14 (13)
N1—C2—N3	115.38 (13)	O4—C4—N4	125.87 (16)
N1—C2—C3	103.30 (12)	O4—C4—N3	126.45 (15)
N3—C2—C3	103.27 (13)	N4—C4—N3	107.68 (14)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WA···Br1^{i}	0.82	2.47	3.2710 (13)	166
O1W—H1WB···O3W^{ii}	0.87	1.93	2.796 (2)	174
O2W—H2WA···O2	0.84	2.02	2.8496 (18)	172
O2W—H2WB···Br1^{iii}	0.90	2.44	3.3397 (15)	177
O3W—H3WA···O1	0.87	1.95	2.8170 (19)	176
O3W—H3WB···Br1	0.85	2.43	3.2728 (14)	175
O4W—H4WC···O3	0.85	2.01	2.663 (5)	133
O4W—H4WC···N10^{iv}	0.85	2.59	3.116 (4)	121
O4W—H4WD···O4^{v}	0.85	2.29	2.765 (4)	115

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x, -y, -z+1$; (iv) $-x+1, -y, -z+2$; (v) $-x+1, -y, -z+1$.

The H atoms of four solvent water molecules were located in a difference Fourier map and refined in their as-found positions relative to the O atoms, with $U_{iso}(H) = 1.2U_{eq}(O)$. The other H atoms were placed in calculated positions, with C—H = 0.95–1.00 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We acknowledge the support of the National Natural Science Foundation of China (NSFC; Nos. 20362003 and 20662003), the International Collaborative Project of the Ministry of Science and Technology (ICPMST; No.

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2003DF000030), the International Collaborative Project of Guizhou Province (ICPGP; No. 2005400101) and the Foundation of the Governor of Guizhou Province.

References

Bruker (2002). *SADABS*, *SAINT* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.

Buschmann, H. J. & Wolff, T. (1999). *J. Photochem. Photobiol. A*, **121**, 99–103.

Day, A. I., Arnold, A. P. & Blanch, R. J. (2000). Int. Patent WO2000 68 232.

Day, A. I., Blanch, R. J., Arnold, A. P., Lorenzo, S., Lewis, G. R. & Dance, I. (2002). *Angew. Chem. Int. Ed.* **41**, 275–277.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Freeman, W. A., Mock, W. L. & Shih, N. Y. (1981). *J. Am. Chem. Soc.* **103**, 7367–7370.

Jon, S. Y., Selvapalam, N., Oh, D. H., Kang, J. K., Kim, S. Y., Jeon, Y. J., Lee, J. W. & Kim, K. (2003). *J. Am. Chem. Soc.* **125**, 10186–10187.

Kim, J., Jung, I. S., Kim, S.-Y., Lee, E., Kang, J.-K., Sakamoto, S., Yamaguchi, K. & Kim, K. (2000). *J. Am. Chem. Soc.* **122**, 540–541.

Lagona, J., Fettinger, C. & Isaacs, L. (2003). *Org. Lett.* **5**, 3745–3747.

Meschke, C., Buschman, H.-J. & Schollmeyer, E. (1999). *Polymer*, **40**, 945–949.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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