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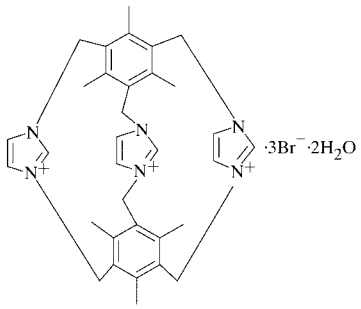
The new imidazolium cyclophane 2,11,13,21,29,31-hexamethyl-5,16,24-triaza-8,19,27-triazoniaheptacyclo[10.10.6.-$1^{1,3}$.$1^{5,8}$.$1^{10,14}$.$1^{16,19}$.$1^{24,27}$]tritriaconta-1,3(29),6,8(30),10,12,-14(31),17,19(32),21,25,27(33)-dodecene tribromide dihydrate, $C_{33}H_{39}N_6^{3+}.3Br^-.2H_2O$, belongs to a type of cylindrical macrobicyclic salt containing imidazolium groups. It has mirror symmetry, the symmetry surface is the plane of the three 2-C atoms of imidazoliums. Three bromine anions are associated with two water molecules by hydrogen bonds. The O—H···Br bond lengths are 2.53 (3) and 2.46 (3) Å.

Comment

The molecular recognition and catalysis properties of cyclophanes have received considerable attention in recent years (Lehn, 1995). It is worthwhile pursuing to design and prepare suitable macrocyclic ligands with fine features. Following our work on imidazole-containing macrocycles (Liu *et al.*, 1999; Luo *et al.*, 1995), for the first time, we synthesize a new type of cyclophane in which two benzene rings are triply bridged by imidazoliums, (I). The compound has C_s symmetry. The parallel capping benzene rings are 5.145 Å apart. The three imidazolium rings are not arranged symmetrically, the angles of them are 54.0, 99.1 and 153.2°, and the distances of the three 2-C atoms of the imidazoliums are 4.703, 4.711 and 4.514 Å. The shape of the compound makes it suitable to form exclusive or inclusive complexes with small anions.



(I)

Different from the case of [1_4]metacyclo-bis-(1,3)-imidazolophanium dichloride dihydrate (Alcalde *et al.*, 1999) ,in each molecule, the three halogen anions form hydrogen bonds with two water molecules rather than with the imidazolophanium. The anions and water molecules fill the space of the imidazolophaniums. It is difficult to point out which bromide anion pairs imidazolium more closely than others. Each group of the three bromides is surrounded by three imidazolophaniums and around each imidazolophanium there are three group of bromides.

Experimental

The title compound was prepared by the dropwise addition of 1,3,5-trimethyl-2,4,6-tris(bromomethyl)benzene to 1,3,5-trimethyl-2,4,6-tri(imidazolymethyl) benzene in highly diluted acetonitrile solutions at 348 K and recrystallized from water.

Crystal data

$C_{33}H_{39}N_6^{3+}\cdot3Br^-\cdot2H_2O$
M_r = 795.46
Monoclinic, $P2_1/m$
a = 9.855 (2) Å
b = 17.306 (3) Å
c = 10.607 (2) Å
β = 114.290 (10)°
V = 1648.9 (5) Å^3
Z = 2
D_x = 1.602 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 3.16–16.44°
μ = 3.710 mm^{-1}
T = 296 (2) K
Flake, colourless
0.56 × 0.40 × 0.28 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: empirical (*SHELXTL*; Siemens, 1994)
T_{min} = 0.227, T_{max} = 0.354
3533 measured reflections
2989 independent reflections
2006 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25°
$h = 0 \rightarrow 11$
$k = -1 \rightarrow 20$
$l = -12 \rightarrow 11$
3 standard reflections every 97 reflections
intensity decay: 5.36%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.073
S = 0.884
2989 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.409 e Å^{-3}
$\Delta\rho_{min}$ = −0.445 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0033 (4)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O—H1A···Br1	0.87 (3)	2.46 (3)	3.326 (4)	173 (5)
O—H1B···Br2	0.83 (2)	2.53 (3)	3.326 (4)	161 (5)

The absorption correction is given by the experimental fact, so it is appropriate. The difference between expected and reported T_{max}/T_{min} may be caused by the fact that the single-crystal is not quite perfect or the measurement of the crystal size is not very precise. The water H atoms H1A and H1B were refined.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS* (Siemens, 1996); data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXTL* (Siemens, 1994).

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References

Alcalde, E., Alvarez-Rúa, C., García-Granda, S., García-Rodriguez, E., Mesuida, N. & Pérez-García, L. (1999). *J. Chem. Soc. Chem. Commun.* pp. 295–296.
Lehn, J.-M. (1995). In *Supramolecular Chemistry*. Weinheim: VCH Publishers.
Liu, Z., Zhou, C., Su, X. & Xie, R. (1999). *Synth. Commun.* **29**, 2979–2983.
Luo, M., Guo, S., Zhou, C. & Xie, R. (1995). *Heterocycles*, 41. 1421–1424.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *XSCANS*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.