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Dicyclohexano-18-crown-6 hydroxonium tribromide

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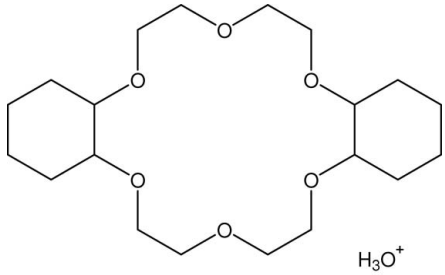
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.009 Å; R factor = 0.043; wR factor = 0.122; data-to-parameter ratio = 20.8.

In the title compound, $C_{20}H_{36}O_6{\cdot}H_3O^+{\cdot}Br_3^-$, the torsion angles of the individual O—C—C—O segments of the macrocyclic strand are ag^+a, ag^-a, ag^+a, ag^-a, ag^+a and ag^-a. Both cyclohexane rings adopt chair conformations. The hydroxonium ion is anchored in the crown-ether cavity by O—H···O hydrogen bonds. In addition, the hydroxonium ion is also involved in O—H···Br hydrogen bonds. The value of the Flack parameter indicates inversion twinning.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related literature, see Harman *et al.* (1976); Nicoló *et al.* (1987); Kusrini (2006); Hassaballa *et al.* (1998); Backer-Dirks *et al.* (1980); Saleh *et al.* (2006). For ring conformations, see: Cremer & Pople (1975).



Br_3^-

Experimental

Crystal data

$C_{20}H_{36}O_6{\cdot}H_3O^+{\cdot}Br_3^-$
M_r = 631.24
Orthorhombic, $P2_12_12_1$
a = 8.2703 (9) Å
b = 13.1939 (14) Å
c = 23.363 (3) Å
V = 2549.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.78 mm^{-1}
T = 100.0 (1) K
0.41 × 0.11 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.248, T_{max} = 0.629
(expected range = 0.233–0.591)
31119 measured reflections
5850 independent reflections
4248 reflections with $I > 2\sigma(I)$
R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.122
S = 1.09
5850 reflections
281 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.87 e Å^{-3}
$\Delta\rho_{min}$ = −0.81 e Å^{-3}
Absolute structure: Flack (1983), with 2535 Friedel pairs
Flack parameter: 0.638 (14)

Table 1
Selected torsion angles (°).

C20—O1—C1—C2	162.0 (5)	C10—O4—C11—C12	−179.1 (4)
C3—O2—C2—C1	−177.9 (5)	C13—O5—C12—C11	−170.8 (4)
O1—C1—C2—O2	66.3 (6)	O4—C11—C12—O5	−60.2 (6)
C2—O2—C3—C4	−179.1 (4)	C12—O5—C13—C14	−177.6 (4)
C5—O3—C4—C3	−179.0 (5)	C15—O6—C14—C13	−178.5 (5)
O2—C3—C4—O3	−69.0 (6)	O5—C13—C14—O6	69.3 (6)
C4—O3—C5—C10	177.1 (5)	C14—O6—C15—C20	176.7 (5)
C4—O3—C5—C6	56.2 (6)	C1—O1—C20—C15	−168.4 (5)
C11—O4—C10—C5	166.1 (5)	O6—C15—C20—O1	−61.0 (6)
O3—C5—C10—O4	61.3 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7O*A*···O3	0.87 (3)	2.05 (3)	2.916 (6)	174 (6)
O7—H7O*A*···O4	0.87 (3)	2.53 (5)	2.960 (6)	111 (5)
O7—H7O*B*···Br3	0.86 (5)	2.61 (4)	3.430 (5)	160 (4)
O7—H7O*C*···O4	0.86 (5)	2.53 (7)	2.960 (6)	112 (5)
O7—H7O*C*···O5	0.86 (5)	2.04 (5)	2.869 (6)	160 (5)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2434).

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supplementary materials

Acta Cryst. (2007). E**63**, o3790-o3791 [**doi:10.1107/S1600536807039244**]

Dicyclohexano-18-crown-6 hydroxonium tribromide

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Comment

The complexation of dicyclohexano-18-crown-6 (DCH18C6) with some lanthanide ions normally leads to the formation of a complex *via* metal–oxygen chelation such as {$La(NO_3)_3(DCH18C6)$]} (Harman *et al.*, 1976) and {$[Eu(NO_3)_2(DCH18C6)]_2[Eu(NO_3)_5]$} (Nicoló *et al.*, 1987) and salt-type compounds namely $[Ln(Pic)_2(DCH18C6)]^+(Pic)^-$ for Ln = La and Ce and also $[Ln(Pic)_2(DCH18C6)]^+(Pic)^-\cdot 0.5(DCH18C6)$ for Ln = Pr and Nd (Kusrini, 2006). However, different products without lanthanide coordination with crown ethers have also been observed namely (15-crown-5)triaquadichlorodioxouranium (Hassaballa *et al.*, 1998) and $[Gd(NO_3)_3(OH_2)_3]$.18-crown-6 (Backer-Dirks *et al.*, 1980).

In our study, no complexation product was obtained from a solution mixture containing dicyclohexano-18-crown-6, europium nitrate and hydrogen bromide. Instead, the formation of a tribromide anion along with a hydroxonium ion occurred. A similar product, namely, bis(2,3-dibromo-6,7,9,10,12,13,15,16-octahydro- 5,8,11,14,17-pentaoxabenzocyclopentadecene)hydroxonium tribromide has been reported (Saleh *et al.*, 2006).

Bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The macrocyclic strand of the molecule displays a series of anti and *gauche* torsion angles for C—O and C—C bonds (Fig. 1). The individual O—C—C—O segments are ag^+a, ag^-a, ag^+a, ag^-a, ag^+a and ag^-a. Relevant torsion angles are listed in Table 1. Both cyclohexane rings adopt chair conformations, with puckering parameters Q = 0.578 (6) Å, θ = 0.0 (6)° and φ = 8(20)° for the C5–C10 ring, and Q = 0.568 (7) Å, θ = 176.2 (7)° and φ = 200 (10)° for the C15–C20 ring (Cremer & Pople, 1975).

The crystal structure is stabilized by O—H···Br and O—H···O hydrogen bonds (Table 2, Fig. 2).

Experimental

The title compound was prepared by the reaction of dicyclohexano-18-crown-6 (0.384 g, 1 mmol) and europium nitrate (0.443 g 1 mmol) in the presence of hydrogen bromide (2 ml, 12.5 *M*, 25 mmol) in 20 ml mixed acetronitrile–methanol (1:1 *v*/*v*) solution. The resulting solution was left to evaporate at room temperature. Orange crystals suitable for X-ray diffraction were collected after 3 weeks (yield 75%; m.p. 430.4–436.8 K). Elemental analysis data: Found (calculated) C 37.17 (38.02) and H 5.55 (6.0)%.

Refinement

The oxonium H atoms were located in a difference Fourier map and refined with O—H and H···H distance restraints of 0.82 (5) Å and 1.37 (1) Å, respectively, and with $U_{iso}(H) = 1.5U_{eq}(O)$. The remaining H atoms were positioned geometrically and treated as riding, with C—H = 0.97–0.98 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$. Because of racemic twinning, the TWIN and BASF instructions were used in the final refinement.

Figures

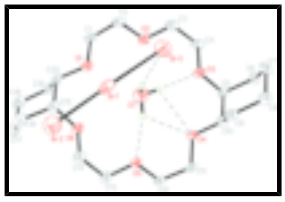

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds are shown as dashed lines.

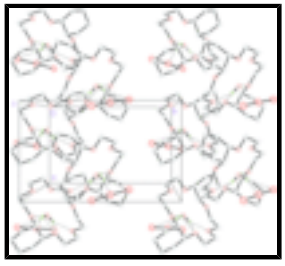

Fig. 2. Part of the crystal packing of the title compound, viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

Dicyclohexano-18-crown-6 hydroxonium tribromide

Crystal data

$C_{20}H_{36}O_6 \cdot H_3O^+ \cdot Br_3^-$	F_{000} = 1280
M_r = 631.24	D_x = 1.645 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5411 reflections
a = 8.2703 (9) Å	θ = 1.7–24.5º
b = 13.1939 (14) Å	μ = 4.78 mm^{-1}
c = 23.363 (3) Å	T = 100.0 (1) K
V = 2549.3 (5) Å^3	Needle, orange
Z = 4	0.41 × 0.11 × 0.11 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	5850 independent reflections
Radiation source: fine-focus sealed tube	4248 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.089
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 27.5º
T = 100.0(1) K	θ_{min} = 1.7º
ω scans	h = −10→10
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −17→17
T_{min} = 0.248, T_{max} = 0.629	l = −30→30
31119 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 1.0999P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rho_{max} = 0.87$ e Å^{-3}
5850 reflections	$\Delta\rho_{min} = -0.81$ e Å^{-3}
281 parameters	Extinction correction: none
6 restraints	Absolute structure: Flack (1983), with 2535 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.638 (14)
Secondary atom site location: difference Fourier map	

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.50581 (7)	0.45651 (5)	0.20640 (2)	0.02836 (15)
Br2	0.46209 (9)	0.57409 (6)	0.28793 (3)	0.0431 (2)
Br3	0.54389 (9)	0.33108 (5)	0.11935 (3)	0.03887 (19)
O1	0.9888 (5)	0.5804 (3)	0.13934 (16)	0.0259 (9)
O2	0.9961 (5)	0.4629 (3)	0.03717 (16)	0.0253 (8)
O3	0.7158 (4)	0.4708 (3)	−0.03546 (17)	0.0230 (9)
O4	0.5259 (4)	0.6398 (3)	−0.02434 (15)	0.0196 (8)
O5	0.5170 (5)	0.7480 (3)	0.07877 (15)	0.0212 (8)
O6	0.7817 (5)	0.7404 (3)	0.15891 (17)	0.0238 (9)
C1	1.1254 (7)	0.5219 (5)	0.1214 (3)	0.0303 (15)
H1A	1.1872	0.5008	0.1546	0.036*
H1B	1.1948	0.5629	0.0973	0.036*
C2	1.0695 (7)	0.4312 (5)	0.0892 (3)	0.0281 (14)

H2A	1.1607	0.3872	0.0810	0.034*
H2B	0.9923	0.3934	0.1120	0.034*
C3	0.9354 (7)	0.3796 (5)	0.0057 (2)	0.0257 (13)
H3A	0.8548	0.3441	0.0282	0.031*
H3B	1.0226	0.3327	−0.0026	0.031*
C4	0.8615 (7)	0.4154 (5)	−0.0490 (3)	0.0282 (14)
H4A	0.9367	0.4588	−0.0693	0.034*
H4B	0.8360	0.3579	−0.0733	0.034*
C5	0.6289 (6)	0.5088 (4)	−0.0847 (2)	0.0193 (12)
H5A	0.6950	0.5600	−0.1041	0.023*
C6	0.5845 (7)	0.4259 (5)	−0.1271 (2)	0.0249 (13)
H6A	0.6821	0.3938	−0.1412	0.030*
H6B	0.5280	0.4554	−0.1594	0.030*
C7	0.4765 (8)	0.3463 (4)	−0.0985 (3)	0.0290 (14)
H7A	0.4443	0.2960	−0.1265	0.035*
H7B	0.5366	0.3121	−0.0685	0.035*
C8	0.3263 (7)	0.3961 (5)	−0.0729 (3)	0.0303 (15)
H8A	0.2601	0.4232	−0.1036	0.036*
H8B	0.2632	0.3453	−0.0529	0.036*
C9	0.3699 (7)	0.4806 (5)	−0.0318 (3)	0.0238 (14)
H9A	0.4249	0.4525	0.0012	0.029*
H9B	0.2721	0.5137	−0.0186	0.029*
C10	0.4794 (7)	0.5582 (4)	−0.0611 (2)	0.0227 (12)
H10A	0.4193	0.5872	−0.0933	0.027*
C11	0.3958 (7)	0.7063 (5)	−0.0104 (3)	0.0261 (14)
H11A	0.3113	0.6686	0.0090	0.031*
H11B	0.3505	0.7344	−0.0453	0.031*
C12	0.4534 (7)	0.7904 (4)	0.0272 (2)	0.0230 (13)
H12A	0.5366	0.8290	0.0078	0.028*
H12B	0.3645	0.8356	0.0361	0.028*
C13	0.5503 (7)	0.8242 (5)	0.1199 (2)	0.0231 (12)
H13A	0.4511	0.8591	0.1301	0.028*
H13B	0.6248	0.8735	0.1040	0.028*
C14	0.6237 (7)	0.7760 (5)	0.1725 (3)	0.0268 (14)
H14A	0.6296	0.8253	0.2032	0.032*
H14B	0.5567	0.7200	0.1852	0.032*
C15	0.8604 (7)	0.6903 (5)	0.2066 (3)	0.0259 (13)
H15A	0.7941	0.6332	0.2196	0.031*
C16	0.8918 (8)	0.7619 (5)	0.2565 (3)	0.0309 (15)
H16A	0.7895	0.7876	0.2707	0.037*
H16B	0.9433	0.7246	0.2874	0.037*
C17	0.9985 (9)	0.8501 (5)	0.2396 (2)	0.0310 (14)
H17A	1.0187	0.8925	0.2727	0.037*
H17B	0.9438	0.8908	0.2109	0.037*
C18	1.1579 (7)	0.8118 (5)	0.2156 (3)	0.0328 (16)
H18A	1.2188	0.7795	0.2461	0.039*
H18B	1.2206	0.8690	0.2020	0.039*
C19	1.1349 (7)	0.7362 (5)	0.1667 (3)	0.0276 (14)
H19A	1.0902	0.7709	0.1337	0.033*

H19B	1.2388	0.7081	0.1558	0.033*
C20	1.0221 (7)	0.6514 (4)	0.1847 (2)	0.0233 (13)
H20A	1.0743	0.6141	0.2160	0.028*
O7	0.6958 (5)	0.5617 (4)	0.0781 (2)	0.0392 (12)
H7OA	0.703 (8)	0.539 (4)	0.0434 (10)	0.059*
H7OB	0.655 (8)	0.512 (3)	0.097 (2)	0.059*
H7OC	0.622 (6)	0.608 (4)	0.077 (3)	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0157 (2)	0.0360 (3)	0.0334 (3)	−0.0008 (3)	−0.0016 (2)	0.0130 (3)
Br2	0.0440 (4)	0.0503 (4)	0.0349 (4)	0.0017 (3)	0.0087 (3)	0.0050 (3)
Br3	0.0446 (4)	0.0289 (3)	0.0431 (4)	−0.0083 (3)	0.0040 (3)	0.0028 (3)
O1	0.0150 (19)	0.024 (2)	0.039 (2)	0.0024 (19)	−0.0083 (18)	−0.0019 (17)
O2	0.0186 (18)	0.025 (2)	0.032 (2)	0.002 (2)	−0.0020 (18)	0.0023 (17)
O3	0.0146 (19)	0.028 (2)	0.026 (2)	0.0076 (18)	0.0023 (16)	0.0007 (19)
O4	0.0102 (19)	0.023 (2)	0.025 (2)	0.0016 (17)	−0.0028 (16)	−0.0025 (15)
O5	0.017 (2)	0.022 (2)	0.0242 (19)	0.0011 (18)	−0.0041 (17)	−0.0007 (15)
O6	0.016 (2)	0.031 (3)	0.025 (2)	0.0010 (18)	−0.0008 (16)	0.0019 (19)
C1	0.012 (3)	0.033 (4)	0.046 (4)	0.009 (3)	−0.007 (3)	0.002 (3)
C2	0.020 (3)	0.026 (3)	0.038 (3)	0.012 (3)	0.001 (3)	0.004 (3)
C3	0.020 (3)	0.023 (3)	0.034 (3)	0.003 (3)	0.007 (3)	0.004 (3)
C4	0.019 (3)	0.029 (4)	0.036 (4)	0.010 (3)	0.010 (3)	−0.002 (3)
C5	0.011 (3)	0.023 (3)	0.025 (3)	−0.005 (2)	0.001 (2)	0.005 (3)
C6	0.021 (3)	0.027 (3)	0.026 (3)	−0.003 (3)	0.004 (2)	−0.004 (3)
C7	0.029 (3)	0.020 (3)	0.038 (3)	−0.003 (3)	0.009 (3)	−0.006 (3)
C8	0.018 (3)	0.029 (4)	0.044 (4)	−0.006 (3)	0.003 (3)	0.000 (3)
C9	0.018 (3)	0.023 (3)	0.030 (3)	0.001 (2)	0.005 (2)	0.002 (3)
C10	0.025 (3)	0.020 (3)	0.024 (3)	0.001 (3)	0.004 (2)	−0.002 (2)
C11	0.013 (3)	0.028 (4)	0.037 (4)	0.005 (3)	−0.004 (3)	0.003 (3)
C12	0.013 (3)	0.027 (3)	0.029 (3)	0.007 (2)	−0.001 (2)	0.000 (2)
C13	0.013 (3)	0.032 (3)	0.024 (3)	0.000 (3)	0.001 (2)	0.001 (3)
C14	0.013 (3)	0.040 (4)	0.028 (3)	−0.001 (3)	0.002 (2)	−0.002 (3)
C15	0.024 (3)	0.026 (3)	0.028 (3)	0.000 (3)	−0.003 (3)	0.006 (3)
C16	0.027 (3)	0.037 (4)	0.029 (3)	−0.006 (3)	−0.005 (3)	0.008 (3)
C17	0.034 (3)	0.035 (4)	0.024 (3)	−0.005 (3)	−0.006 (3)	0.000 (2)
C18	0.022 (3)	0.036 (4)	0.040 (4)	−0.007 (3)	−0.009 (3)	0.005 (3)
C19	0.018 (3)	0.030 (4)	0.035 (4)	−0.003 (3)	−0.005 (3)	0.007 (3)
C20	0.018 (3)	0.024 (3)	0.028 (3)	−0.003 (3)	−0.009 (3)	0.006 (2)
O7	0.033 (2)	0.044 (3)	0.041 (3)	0.002 (2)	0.004 (2)	0.002 (2)

Geometric parameters (Å, °)

Br1—Br2	2.4831 (9)	C8—H8B	0.97
Br1—Br3	2.6408 (9)	C9—C10	1.528 (8)
O1—C1	1.430 (7)	C9—H9A	0.97
O1—C20	1.442 (7)	C9—H9B	0.97
O2—C3	1.415 (7)	C10—H10A	0.98

O2—C2	1.422 (7)	C11—C12	1.494 (8)
O3—C4	1.444 (7)	C11—H11A	0.97
O3—C5	1.446 (7)	C11—H11B	0.97
O4—C11	1.427 (7)	C12—H12A	0.97
O4—C10	1.429 (6)	C12—H12B	0.97
O5—C13	1.418 (7)	C13—C14	1.511 (8)
O5—C12	1.428 (7)	C13—H13A	0.97
O6—C14	1.425 (7)	C13—H13B	0.97
O6—C15	1.450 (7)	C14—H14A	0.97
C1—C2	1.488 (9)	C14—H14B	0.97
C1—H1A	0.97	C15—C20	1.521 (8)
C1—H1B	0.97	C15—C16	1.522 (9)
C2—H2A	0.97	C15—H15A	0.98
C2—H2B	0.97	C16—C17	1.513 (9)
C3—C4	1.492 (8)	C16—H16A	0.97
C3—H3A	0.97	C16—H16B	0.97
C3—H3B	0.97	C17—C18	1.519 (9)
C4—H4A	0.97	C17—H17A	0.97
C4—H4B	0.97	C17—H17B	0.97
C5—C10	1.503 (8)	C18—C19	1.530 (9)
C5—C6	1.521 (8)	C18—H18A	0.97
C5—H5A	0.98	C18—H18B	0.97
C6—C7	1.531 (8)	C19—C20	1.518 (8)
C6—H6A	0.97	C19—H19A	0.97
C6—H6B	0.97	C19—H19B	0.97
C7—C8	1.527 (8)	C20—H20A	0.98
C7—H7A	0.97	O7—H7OA	0.87 (3)
C7—H7B	0.97	O7—H7OB	0.86 (5)
C8—C9	1.516 (9)	O7—H7OC	0.86 (5)
C8—H8A	0.97		
Br2—Br1—Br3	178.48 (4)	C5—C10—H10A	107.7
C1—O1—C20	114.5 (4)	C9—C10—H10A	107.7
C3—O2—C2	111.6 (4)	O4—C11—C12	110.5 (5)
C4—O3—C5	114.6 (4)	O4—C11—H11A	109.5
C11—O4—C10	113.4 (4)	C12—C11—H11A	109.5
C13—O5—C12	111.4 (4)	O4—C11—H11B	109.5
C14—O6—C15	113.0 (4)	C12—C11—H11B	109.5
O1—C1—C2	109.7 (5)	H11A—C11—H11B	108.1
O1—C1—H1A	109.7	O5—C12—C11	108.9 (5)
C2—C1—H1A	109.7	O5—C12—H12A	109.9
O1—C1—H1B	109.7	C11—C12—H12A	109.9
C2—C1—H1B	109.7	O5—C12—H12B	109.9
H1A—C1—H1B	108.2	C11—C12—H12B	109.9
O2—C2—C1	109.2 (5)	H12A—C12—H12B	108.3
O2—C2—H2A	109.8	O5—C13—C14	109.3 (5)
C1—C2—H2A	109.8	O5—C13—H13A	109.8
O2—C2—H2B	109.8	C14—C13—H13A	109.8
C1—C2—H2B	109.8	O5—C13—H13B	109.8
H2A—C2—H2B	108.3	C14—C13—H13B	109.8

O2—C3—C4	110.2 (5)	H13A—C13—H13B	108.3
O2—C3—H3A	109.6	O6—C14—C13	109.0 (5)
C4—C3—H3A	109.6	O6—C14—H14A	109.9
O2—C3—H3B	109.6	C13—C14—H14A	109.9
C4—C3—H3B	109.6	O6—C14—H14B	109.9
H3A—C3—H3B	108.1	C13—C14—H14B	109.9
O3—C4—C3	108.3 (5)	H14A—C14—H14B	108.3
O3—C4—H4A	110.0	O6—C15—C20	106.8 (5)
C3—C4—H4A	110.0	O6—C15—C16	112.5 (5)
O3—C4—H4B	110.0	C20—C15—C16	108.5 (5)
C3—C4—H4B	110.0	O6—C15—H15A	109.6
H4A—C4—H4B	108.4	C20—C15—H15A	109.6
O3—C5—C10	105.5 (4)	C16—C15—H15A	109.6
O3—C5—C6	112.8 (5)	C17—C16—C15	112.0 (5)
C10—C5—C6	110.6 (4)	C17—C16—H16A	109.2
O3—C5—H5A	109.3	C15—C16—H16A	109.2
C10—C5—H5A	109.3	C17—C16—H16B	109.2
C6—C5—H5A	109.3	C15—C16—H16B	109.2
C5—C6—C7	110.5 (5)	H16A—C16—H16B	107.9
C5—C6—H6A	109.5	C16—C17—C18	110.3 (5)
C7—C6—H6A	109.5	C16—C17—H17A	109.6
C5—C6—H6B	109.5	C18—C17—H17A	109.6
C7—C6—H6B	109.5	C16—C17—H17B	109.6
H6A—C6—H6B	108.1	C18—C17—H17B	109.6
C8—C7—C6	110.5 (5)	H17A—C17—H17B	108.1
C8—C7—H7A	109.6	C17—C18—C19	112.6 (5)
C6—C7—H7A	109.6	C17—C18—H18A	109.1
C8—C7—H7B	109.6	C19—C18—H18A	109.1
C6—C7—H7B	109.6	C17—C18—H18B	109.1
H7A—C7—H7B	108.1	C19—C18—H18B	109.1
C9—C8—C7	111.8 (5)	H18A—C18—H18B	107.8
C9—C8—H8A	109.3	C20—C19—C18	110.4 (5)
C7—C8—H8A	109.3	C20—C19—H19A	109.6
C9—C8—H8B	109.3	C18—C19—H19A	109.6
C7—C8—H8B	109.3	C20—C19—H19B	109.6
H8A—C8—H8B	107.9	C18—C19—H19B	109.6
C8—C9—C10	110.5 (5)	H19A—C19—H19B	108.1
C8—C9—H9A	109.5	O1—C20—C19	113.1 (5)
C10—C9—H9A	109.5	O1—C20—C15	107.4 (4)
C8—C9—H9B	109.5	C19—C20—C15	112.6 (5)
C10—C9—H9B	109.5	O1—C20—H20A	107.8
H9A—C9—H9B	108.1	C19—C20—H20A	107.8
O4—C10—C5	109.0 (4)	C15—C20—H20A	107.8
O4—C10—C9	113.3 (4)	H7OA—O7—H7OB	104 (5)
C5—C10—C9	111.2 (5)	H7OA—O7—H7OC	105 (6)
O4—C10—H10A	107.7	H7OB—O7—H7OC	106 (6)
C20—O1—C1—C2	162.0 (5)	C10—O4—C11—C12	−179.1 (4)
C3—O2—C2—C1	−177.9 (5)	C13—O5—C12—C11	−170.8 (4)
O1—C1—C2—O2	66.3 (6)	O4—C11—C12—O5	−60.2 (6)

C2—O2—C3—C4	−179.1 (4)	C12—O5—C13—C14	−177.6 (4)
C5—O3—C4—C3	−179.0 (5)	C15—O6—C14—C13	−178.5 (5)
O2—C3—C4—O3	−69.0 (6)	O5—C13—C14—O6	69.3 (6)
C4—O3—C5—C10	177.1 (5)	C14—O6—C15—C20	176.7 (5)
C4—O3—C5—C6	56.2 (6)	C14—O6—C15—C16	−64.3 (6)
O3—C5—C6—C7	60.0 (6)	O6—C15—C16—C17	−59.5 (7)
C10—C5—C6—C7	−57.9 (6)	C20—C15—C16—C17	58.5 (7)
C5—C6—C7—C8	56.1 (7)	C15—C16—C17—C18	−57.3 (7)
C6—C7—C8—C9	−55.2 (7)	C16—C17—C18—C19	53.9 (7)
C7—C8—C9—C10	55.0 (7)	C17—C18—C19—C20	−52.6 (7)
C11—O4—C10—C5	166.1 (5)	C1—O1—C20—C19	66.7 (6)
C11—O4—C10—C9	−69.6 (6)	C1—O1—C20—C15	−168.4 (5)
O3—C5—C10—O4	61.3 (5)	C18—C19—C20—O1	176.9 (5)
C6—C5—C10—O4	−176.4 (4)	C18—C19—C20—C15	54.9 (6)
O3—C5—C10—C9	−64.3 (6)	O6—C15—C20—O1	−61.0 (6)
C6—C5—C10—C9	58.1 (6)	C16—C15—C20—O1	177.4 (4)
C8—C9—C10—O4	−179.6 (5)	O6—C15—C20—C19	64.1 (6)
C8—C9—C10—C5	−56.4 (6)	C16—C15—C20—C19	−57.4 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7OA···O3	0.87 (3)	2.05 (3)	2.916 (6)	174 (6)
O7—H7OA···O4	0.87 (3)	2.53 (5)	2.960 (6)	111 (5)
O7—H7OB···Br3	0.86 (5)	2.61 (4)	3.430 (5)	160 (4)
O7—H7OC···O4	0.86 (5)	2.53 (7)	2.960 (6)	112 (5)
O7—H7OC···O5	0.86 (5)	2.04 (5)	2.869 (6)	160 (5)

Fig. 1

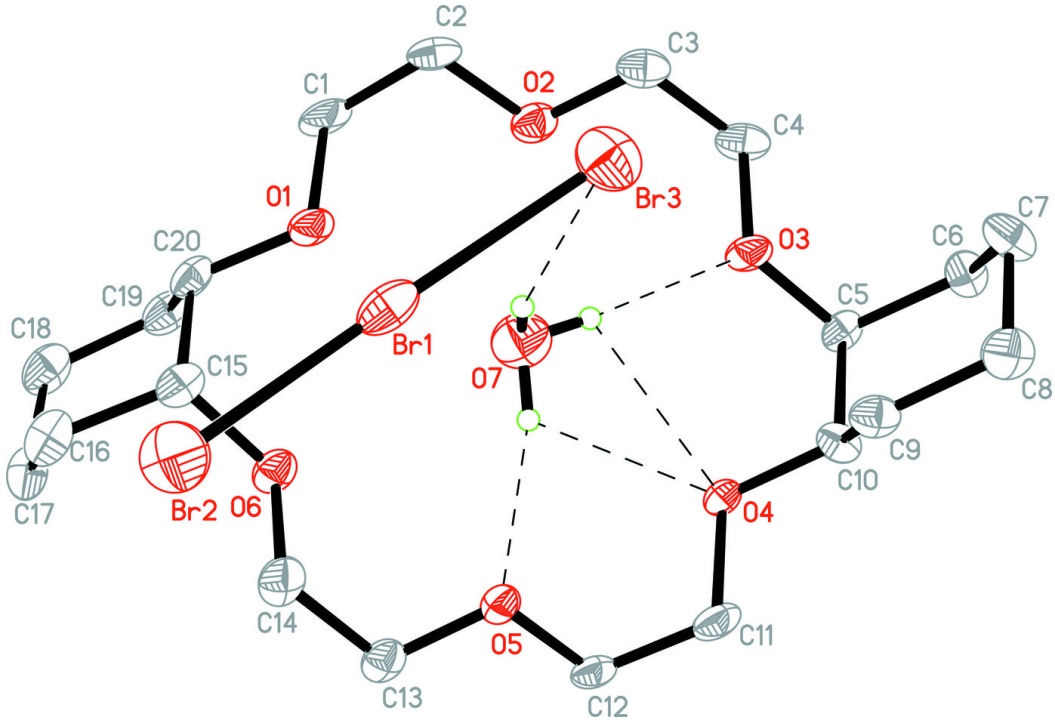
C2
C3
C1
O2
C4
O1
Br3
C20
O3
C7
C6
C19
C5
C18
Br1
O7
C15
C8
C16
C9
C10
C17
Br2
O6
O4
C14
O5
C11
C13
C12

Fig. 2