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2,17-Dibromo-6,7,9,10,12,13-hexahydrodibenzo[*b*,*e*][1,4,7,10,13]pentaoxacyclopentadecin monohydrate

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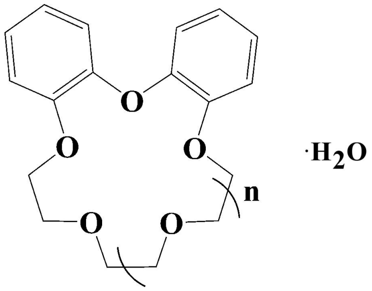
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.053; wR factor = 0.156; data-to-parameter ratio = 17.9.

In the title compound, $C_{18}H_{18}Br_2O_5\cdot H_2O$, the solvent water molecule is bonded to two O atoms of a crown ether molecule by O—H···O hydrogen bonds. In the crystal structure, molecules of this macrocycle form columns along the [100] crystallographic direction as a result of stacking interactions between aromatic rings (centroid–centroid distance 3.5 Å).

Related literature

For related literature, see: Gokel & Korzeniowski (1982); Hiraoka (1982); Kamalov *et al.* (2003); Zefirov & Zorky (1995).



Experimental

Crystal data

$C_{18}H_{18}Br_2O_5\cdot H_2O$	V = 1853.8 (16) Å^3
M_r = 492.16	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 10.012 (5) Å	μ = 4.41 mm^{-1}
b = 26.850 (5) Å	T = 100 (2) K
c = 7.031 (5) Å	0.80 × 0.10 × 0.10 mm
β = 101.245 (5)°	

Data collection

Oxford Diffraction Xcalibur 3 diffractometer	8042 measured reflections
Absorption correction: analytical (Alcock, 1970)	4196 independent reflections
$T_{\min}$ = 0.267, $T_{\max}$ = 0.674	2786 reflections with $I > 2\sigma(I)$
	$R_{\rm int}$ = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053	235 parameters
$wR(F^2)$ = 0.156	H-atom parameters constrained
S = 1.03	$\Delta\rho_{\max}$ = 1.56 e Å^{-3}
4196 reflections	$\Delta\rho_{\min}$ = −1.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1S—H1OA···O3	0.82	2.16	2.968 (5)	172
O1S—H1OB···O5	1.02	2.21	3.125 (5)	150

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2381).

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supplementary materials

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2,17-Dibromo-6,7,9,10,12,13-hexahydrodibenzo[*b*,*e*][1,4,7,10,13]pentaoxacyclopentadecin monohydrate

S. A. Kotlyar, S. V. Shishkina, O. V. Shishkin, R. Y. Grygorash, S. M. Pluzhnik-Gladyr and G. L. Kamalov

Comment

Crown ethers (CE), containing diphenyl oxide fragment (I, n=1–3), are structural isomers of the corresponding distal ("symmertic", $x=y$ if n=2) and proximal ("asymmetric", $x \neq y$ if n=1–3) dibenzoCE (II) (Fig. 3). Products of disubstitution in the aromatic fragment of compounds (I) were not obtained before. As known, dibenzoCE of type (II) in such reactions form only the mixture of *cis*- and *trans*-isomers (Hiraoka, 1982; Gokel & Korzeniowski, 1982), which can be very rarely separated. We found out (Kamalov *et al.*, 2003) that bromination of [1.4]dibenzo-15-crown-5 (III) by *N*-bromosuccinimide leads only to $5,5^{I}$-dibromide (IV) (Fig. 4). In the case of other CE (I) under investigation the bromination also takes place selectively. In this paper we report the crystal structure of the compound (IV) monohydrate (Fig. 1), which is formed during treatment of the obtained CE with water. Analysis of the molecular structure of the title compound demonstrates that the O4, O5, O1 and O2 atoms are co-planar within 0.03 Å. The O3 atom is displaced from this plane by 0.76 Å. The O—C—C—O fragments have alternating +*sc* and -*sc* conformations (Table 1). The aromatic rings are rotated relative to each other (angle between their mean planes is 65.8 °) due to repulsion between them (there are shortened intramolecular contacts of H5···C7 = 2.62 Å [sum of van der Waals radii is 2.87 Å (Zefirov & Zorky, 1995)], H5···C8 = 2.76 Å, C5···C8 3.24 Å [sum of van der Waals radii is 3.42 Å]). The O1 and O2 atoms are displaced from the plane of aromatic ring (the O1—C7—C12—O2 torsion angle is 8.7 (7) °). In the crystal structure, molecules of compound (IV) form columns along the (0 0 1) crystallographic direction with parallel arrangement of aromatic rings (Fig.2). The distance between neighbouring phenyl rings in columns is *ca* 3.5 Å, and it allows us to assume the existence of π···π stacking interactions. The macrocycle cavity is capped from one side by water molecule, which is bonded with two oxygen atoms of macrocycle by intermolecular hydrogen bonds (see Table 1). The other side of cavity is capped by a phenyl ring of a symmetry related molecule. A weak intermolecular hydrogen bond C3—H3···O2(x,$0.5-y$,$1/2+z$) with H···O = 2.42 Å, C—H···O = 163 ° is also present. Some short intermolecular distances are also observed, *e.g.* Br1···H16*b*($x-1$,$0.5-y$,$1/2+z$) = 3.09 Å (3.23 Å), Br2···Br(2($-x$,$1-y$,$2-z$) = 3.73 Å (3.94 Å), Br2···H15*b*($x-1$,y,$1+z$) = 3.08 Å (3.23 Å).

Experimental

N-Bromosuccinimide (1.9 g, 10.68 mmol) was added to a solution of [1.4]dibenzo-15-crown-5 (1.63 g, 5.15 mmol) in 15 ml $CHCl_3$. The reaction mixture was refluxed for 1 h then cooled and filtered. The residue was washed with 15 ml of $CHCl_3$ and the combined filtrates were evaporated at the lowered pressure until dry. The was residue was washed with water, filtered, dried at the open air to constant weight and crystallized from propanol-2. The yield of compound IV is 83% (2.03 g, white crystals), [m.p. 375–376 K]. Analysis, calculated for $C_{18}H_{18}Br_2O_5$: C 45.52, H 3.79, Br 33.68%; found: C 45.60, H 3.83, Br 33.70%. The crystals are soluble in benzene, acetone, dichloromethane, chloroform, dimethyl sulfoxide, dimethyl formamide and other organic solvents. ^{1}H NMR spectrum (Varian VXR-300, in $CDCl_3$, relative to the inner standard Me_4Si): H_{ar} 7.22–7.20 (2*H*, dd), J=9.3, J=1.5; 7.06–7.03 (2*H*, d), J=9.3; 6.87–7.63 (2*H*, d), J=1.5; CH_2CH_2O- 4.18–4.15 (4*H*, m), 3.64–3.61 (4*H*, m), 3.52 (4*H*, s). TLC (Silufol UV 254, Kavalier, plates, the visualization was performed under UV light): R_f 0.54 (aceton-n-hexane = 1:1). Transparent colourless crystals of (IV)$\cdot H_2O$, suitable for X-ray were obtained by spontaneous evaporation of 95% ethanol solution.

Refinement

All hydrogen atoms were located in electron density difference maps but included in the refinement in the riding-model approximation with C—H = 0.95–0.99Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms of the water molecule were included in the refinement in their as found positions with $U_{iso}(H) = 1.5U_{eq}(C)$. The three largest peaks on the final difference Fourier are 1.12, 0.79 and 1.87Å from C17, C11 and O1s, respectively.

Figures

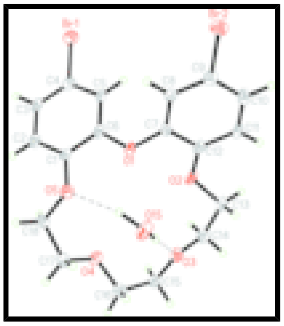


Fig. 1. The molecular structure of (I) with the atomic membering. All atoms are shown with displacement ellipsoids drawn at the 50% probability level. The dashed lines indicate hydrogen bonds.

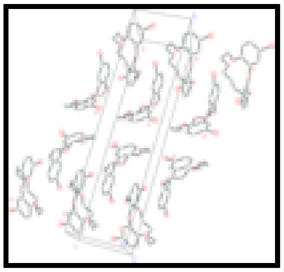


Fig. 2. Part of the crystal structure of the title compound.

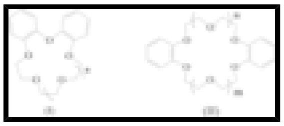


Fig. 3. The structures of (I) and (II).

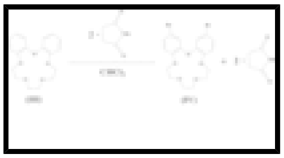


Fig. 4. The formation of (IV).

2,17-Dibromo-6,7,9,10,12,13- hexahydrodibenzo[b,e][1,4,7,10,13]pentaoxacyclopentadecin monohydrate

Crystal data

$C_{18}H_{18}Br_2O_5\cdot H_2O$	$F_{000} = 984$
$M_r = 492.16$	$D_x = 1.763$ Mg m^{-3}

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

a = 10.012 (5) Å

b = 26.850 (5) Å

c = 7.031 (5) Å

β = 101.245 (5)°

V = 1853.8 (16) Å^3

Z = 4

Mo $K\alpha$ radiation
λ = 0.71069 Å

Cell parameters from 3187 reflections

θ = 4–35°

μ = 4.41 mm^{-1}

T = 100 (2) K

Needle, colourless

0.80 × 0.10 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur 3
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

T = 100(2) K

ω scans

Absorption correction: analytical
(Alcock, 1970)

$T_{\min}$ = 0.267, $T_{\max}$ = 0.674

8042 measured reflections

4196 independent reflections

2786 reflections with $I > 2\sigma(I)$

R_{int} = 0.049

$\theta_{\max}$ = 27.5°

$\theta_{\min}$ = 3.1°

$h = -13 \rightarrow 12$

$k = -34 \rightarrow 20$

$l = -9 \rightarrow 7$

2 standard reflections

every 50 reflections

intensity decay: ?

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.053

$wR(F^2)$ = 0.156

S = 1.03

4196 reflections

235 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max}$ = 0.001

$\Delta\rho_{\max}$ = 1.56 e Å^{-3}

$\Delta\rho_{\min}$ = −1.40 e Å^{-3}

Extinction correction: none

Special details

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 > 2sigma(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	0.17500 (5)	0.20334 (2)	0.59174 (7)	0.02721 (18)
Br2	0.09743 (5)	0.45440 (2)	0.88064 (7)	0.02732 (18)
O1	0.4996 (3)	0.36064 (14)	0.6514 (5)	0.0253 (8)
O2	0.4725 (4)	0.41181 (13)	0.3243 (5)	0.0241 (8)
O3	0.7321 (3)	0.42023 (14)	0.2538 (5)	0.0214 (7)
O4	0.8663 (3)	0.33494 (13)	0.4418 (5)	0.0218 (8)
O5	0.7177 (3)	0.30854 (14)	0.7383 (5)	0.0223 (8)
C1	0.5977 (5)	0.28185 (19)	0.7094 (6)	0.0172 (10)
C2	0.5905 (5)	0.2310 (2)	0.7246 (7)	0.0206 (10)
H2	0.6713	0.2118	0.7584	0.025*
C3	0.4628 (5)	0.20763 (19)	0.6897 (6)	0.0196 (10)
H3	0.4565	0.1725	0.6998	0.024*
C4	0.3470 (5)	0.2359 (2)	0.6409 (6)	0.0199 (10)
C5	0.3525 (5)	0.28774 (19)	0.6283 (6)	0.0177 (10)
H5	0.2715	0.3069	0.5954	0.021*
C6	0.4782 (5)	0.31043 (19)	0.6646 (6)	0.0190 (10)
C7	0.3927 (5)	0.39384 (19)	0.6106 (7)	0.0194 (10)
C8	0.3062 (5)	0.4028 (2)	0.7389 (7)	0.0193 (10)
H8	0.3116	0.3827	0.8514	0.023*
C9	0.2113 (5)	0.4413 (2)	0.7021 (7)	0.0215 (11)
C10	0.2026 (5)	0.4705 (2)	0.5394 (7)	0.0223 (11)
H10	0.1378	0.4967	0.5159	0.027*
C11	0.2878 (5)	0.46175 (18)	0.4100 (7)	0.0204 (10)
H11	0.2805	0.4819	0.2974	0.024*
C12	0.3843 (5)	0.42354 (19)	0.4431 (7)	0.0215 (10)
C13	0.5051 (5)	0.44934 (19)	0.1948 (7)	0.0213 (11)
H13B	0.5351	0.4803	0.2670	0.026*
H13A	0.4244	0.4570	0.0933	0.026*
C14	0.6174 (5)	0.4288 (2)	0.1053 (7)	0.0244 (11)
H14B	0.5879	0.3972	0.0372	0.029*
H14A	0.6408	0.4527	0.0097	0.029*
C15	0.8397 (5)	0.3949 (2)	0.1904 (7)	0.0246 (11)
H15B	0.8895	0.4180	0.1193	0.029*
H15A	0.8032	0.3672	0.1026	0.029*
C16	0.9332 (5)	0.3751 (2)	0.3668 (7)	0.0249 (11)
H16B	1.0191	0.3634	0.3323	0.030*
H16A	0.9552	0.4017	0.4655	0.030*
C17	0.9287 (5)	0.3213 (2)	0.6340 (7)	0.0211 (10)
H17B	0.9388	0.3509	0.7196	0.025*
H17A	1.0201	0.3071	0.6358	0.025*
C18	0.8378 (5)	0.2831 (2)	0.7032 (7)	0.0211 (10)
H18B	0.8119	0.2569	0.6035	0.025*

H18A	0.8864	0.2671	0.8239	0.025*
O1S	0.7389 (4)	0.42336 (15)	0.6775 (5)	0.0303 (9)
H1OA	0.7401	0.4253	0.5621	0.046*
H1OB	0.7213	0.3907	0.7409	0.046*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0280 (3)	0.0202 (3)	0.0331 (3)	−0.0049 (2)	0.0050 (2)	−0.0027 (2)
Br2	0.0275 (3)	0.0268 (3)	0.0303 (3)	0.0004 (2)	0.0121 (2)	−0.0046 (2)
O1	0.0239 (18)	0.0122 (18)	0.041 (2)	0.0017 (15)	0.0104 (15)	0.0093 (16)
O2	0.0338 (19)	0.0107 (18)	0.0316 (19)	−0.0005 (16)	0.0159 (15)	0.0015 (15)
O3	0.0257 (18)	0.0184 (19)	0.0205 (17)	0.0036 (16)	0.0059 (13)	0.0013 (14)
O4	0.0285 (18)	0.0183 (19)	0.0183 (16)	−0.0032 (16)	0.0042 (13)	0.0008 (14)
O5	0.0242 (18)	0.0177 (19)	0.0265 (17)	0.0012 (15)	0.0084 (14)	−0.0010 (15)
C1	0.021 (2)	0.019 (3)	0.014 (2)	−0.002 (2)	0.0071 (17)	0.0030 (19)
C2	0.026 (2)	0.016 (3)	0.021 (2)	0.004 (2)	0.0082 (19)	0.001 (2)
C3	0.031 (3)	0.011 (2)	0.018 (2)	−0.001 (2)	0.0091 (19)	0.0009 (19)
C4	0.024 (2)	0.018 (3)	0.018 (2)	−0.004 (2)	0.0043 (18)	−0.002 (2)
C5	0.021 (2)	0.016 (3)	0.017 (2)	0.001 (2)	0.0060 (17)	0.0037 (19)
C6	0.030 (3)	0.012 (2)	0.017 (2)	0.004 (2)	0.0094 (19)	−0.0003 (19)
C7	0.018 (2)	0.015 (3)	0.025 (2)	0.003 (2)	0.0042 (18)	0.002 (2)
C8	0.018 (2)	0.020 (3)	0.020 (2)	−0.001 (2)	0.0038 (17)	0.002 (2)
C9	0.024 (3)	0.017 (3)	0.024 (2)	−0.004 (2)	0.0042 (19)	−0.005 (2)
C10	0.024 (2)	0.014 (3)	0.026 (3)	0.001 (2)	−0.002 (2)	−0.004 (2)
C11	0.027 (3)	0.008 (2)	0.026 (2)	−0.001 (2)	0.005 (2)	0.000 (2)
C12	0.028 (3)	0.013 (3)	0.024 (2)	−0.002 (2)	0.0075 (19)	−0.001 (2)
C13	0.030 (3)	0.012 (3)	0.021 (2)	0.001 (2)	0.005 (2)	0.003 (2)
C14	0.033 (3)	0.020 (3)	0.022 (2)	0.003 (2)	0.008 (2)	−0.002 (2)
C15	0.030 (3)	0.020 (3)	0.026 (3)	−0.001 (2)	0.013 (2)	−0.001 (2)
C16	0.024 (2)	0.024 (3)	0.027 (3)	0.000 (2)	0.007 (2)	−0.002 (2)
C17	0.023 (2)	0.019 (3)	0.022 (2)	0.003 (2)	0.0047 (19)	−0.002 (2)
C18	0.021 (2)	0.021 (3)	0.024 (2)	0.007 (2)	0.0097 (18)	0.002 (2)
O1S	0.047 (2)	0.019 (2)	0.0265 (19)	−0.0048 (18)	0.0104 (16)	0.0002 (16)

Geometric parameters (Å, °)

Br1—C4	1.901 (5)	C8—H8	0.9500
Br2—C9	1.886 (5)	C9—C10	1.376 (7)
O1—C6	1.371 (6)	C10—C11	1.383 (7)
O1—C7	1.379 (6)	C10—H10	0.9500
O2—C12	1.365 (6)	C11—C12	1.398 (7)
O2—C13	1.438 (6)	C11—H11	0.9500
O3—C14	1.412 (6)	C13—C14	1.498 (7)
O3—C15	1.418 (6)	C13—H13B	0.9900
O4—C17	1.421 (6)	C13—H13A	0.9900
O4—C16	1.424 (6)	C14—H14B	0.9900
O5—C1	1.380 (6)	C14—H14A	0.9900
O5—C18	1.446 (6)	C15—C16	1.497 (7)

C1—C2	1.373 (7)	C15—H15B	0.9900
C1—C6	1.404 (7)	C15—H15A	0.9900
C2—C3	1.402 (7)	C16—H16B	0.9900
C2—H2	0.9500	C16—H16A	0.9900
C3—C4	1.371 (7)	C17—C18	1.514 (7)
C3—H3	0.9500	C17—H17B	0.9900
C4—C5	1.398 (7)	C17—H17A	0.9900
C5—C6	1.376 (7)	C18—H18B	0.9900
C5—H5	0.9500	C18—H18A	0.9900
C7—C8	1.388 (6)	O1S—H1OA	0.8159
C7—C12	1.411 (7)	O1S—H1OB	1.0151
C8—C9	1.393 (7)		

C6—O1—C7	121.6 (4)	O2—C12—C7	116.1 (4)
C12—O2—C13	118.7 (4)	C11—C12—C7	118.7 (5)
C14—O3—C15	113.8 (4)	O2—C13—C14	106.8 (4)
C17—O4—C16	113.3 (4)	O2—C13—H13B	110.4
C1—O5—C18	117.3 (4)	C14—C13—H13B	110.4
C2—C1—O5	124.2 (4)	O2—C13—H13A	110.4
C2—C1—C6	120.4 (5)	C14—C13—H13A	110.4
O5—C1—C6	115.4 (5)	H13B—C13—H13A	108.6
C1—C2—C3	119.4 (5)	O3—C14—C13	108.5 (4)
C1—C2—H2	120.3	O3—C14—H14B	110.0
C3—C2—H2	120.3	C13—C14—H14B	110.0
C4—C3—C2	119.6 (5)	O3—C14—H14A	110.0
C4—C3—H3	120.2	C13—C14—H14A	110.0
C2—C3—H3	120.2	H14B—C14—H14A	108.4
C3—C4—C5	121.7 (5)	O3—C15—C16	107.5 (4)
C3—C4—Br1	118.9 (4)	O3—C15—H15B	110.2
C5—C4—Br1	119.4 (4)	C16—C15—H15B	110.2
C6—C5—C4	118.4 (5)	O3—C15—H15A	110.2
C6—C5—H5	120.8	C16—C15—H15A	110.2
C4—C5—H5	120.8	H15B—C15—H15A	108.5
O1—C6—C5	124.9 (5)	O4—C16—C15	108.2 (4)
O1—C6—C1	114.5 (4)	O4—C16—H16B	110.1
C5—C6—C1	120.5 (5)	C15—C16—H16B	110.1
O1—C7—C8	122.3 (4)	O4—C16—H16A	110.1
O1—C7—C12	117.1 (4)	C15—C16—H16A	110.1
C8—C7—C12	120.1 (5)	H16B—C16—H16A	108.4
C7—C8—C9	119.8 (4)	O4—C17—C18	107.4 (4)
C7—C8—H8	120.1	O4—C17—H17B	110.2
C9—C8—H8	120.1	C18—C17—H17B	110.2
C10—C9—C8	120.5 (5)	O4—C17—H17A	110.2
C10—C9—Br2	119.9 (4)	C18—C17—H17A	110.2
C8—C9—Br2	119.6 (4)	H17B—C17—H17A	108.5
C9—C10—C11	120.2 (5)	O5—C18—C17	107.8 (4)
C9—C10—H10	119.9	O5—C18—H18B	110.1
C11—C10—H10	119.9	C17—C18—H18B	110.1
C10—C11—C12	120.7 (5)	O5—C18—H18A	110.1
C10—C11—H11	119.7	C17—C18—H18A	110.1

C12—C11—H11	119.7	H18B—C18—H18A	108.5
O2—C12—C11	125.2 (4)	H1OA—O1S—H1OB	121.9

C18—O5—C1—C2	26.5 (6)	C7—C8—C9—Br2	−178.3 (4)
C18—O5—C1—C6	−154.4 (4)	C8—C9—C10—C11	0.3 (7)
O5—C1—C2—C3	−179.2 (4)	Br2—C9—C10—C11	178.6 (4)
C6—C1—C2—C3	1.7 (7)	C9—C10—C11—C12	−0.5 (7)
C1—C2—C3—C4	0.0 (7)	C13—O2—C12—C11	22.8 (7)
C2—C3—C4—C5	−1.2 (7)	C13—O2—C12—C7	−157.8 (4)
C2—C3—C4—Br1	179.4 (3)	C10—C11—C12—O2	179.7 (5)
C3—C4—C5—C6	0.6 (7)	C10—C11—C12—C7	0.4 (7)
Br1—C4—C5—C6	180.0 (3)	O1—C7—C12—O2	8.4 (7)
C7—O1—C6—C5	4.1 (7)	C8—C7—C12—O2	−179.4 (4)
C7—O1—C6—C1	−179.2 (4)	O1—C7—C12—C11	−172.2 (4)
C4—C5—C6—O1	177.7 (4)	C8—C7—C12—C11	0.0 (7)
C4—C5—C6—C1	1.3 (6)	C12—O2—C13—C14	172.0 (4)
C2—C1—C6—O1	−179.2 (4)	C15—O3—C14—C13	172.5 (4)
O5—C1—C6—O1	1.7 (6)	O2—C13—C14—O3	−62.3 (5)
C2—C1—C6—C5	−2.4 (7)	C14—O3—C15—C16	−163.2 (4)
O5—C1—C6—C5	178.5 (4)	C17—O4—C16—C15	−165.0 (4)
C6—O1—C7—C8	67.1 (6)	O3—C15—C16—O4	71.5 (5)
C6—O1—C7—C12	−120.8 (5)	C16—O4—C17—C18	173.1 (4)
O1—C7—C8—C9	171.6 (5)	C1—O5—C18—C17	149.0 (4)
C12—C7—C8—C9	−0.2 (7)	O4—C17—C18—O5	−72.2 (5)
C7—C8—C9—C10	0.1 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1S—H1OA···O3	0.82	2.16	2.968 (5)	172
O1S—H1OB···O5	1.02	2.21	3.125 (5)	150

Fig. 1

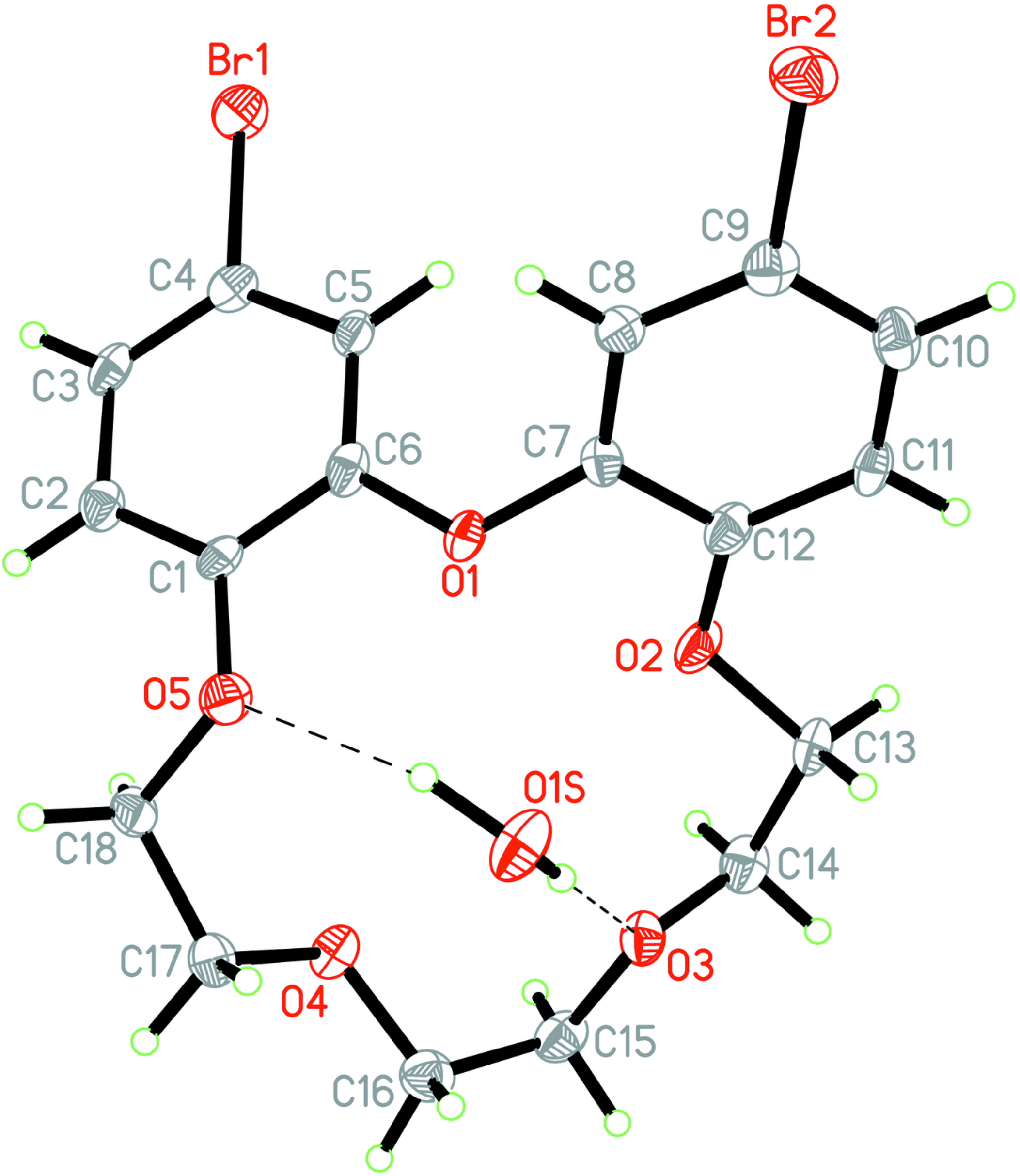

Br1
Br2
C9
C4
C5
C8
C3
C10
C6
C7
C11
C2
C12
C1
O1
O2
O5
C13
O1S
C18
C14
C17
O3
O4
C15
C16

Fig. 2

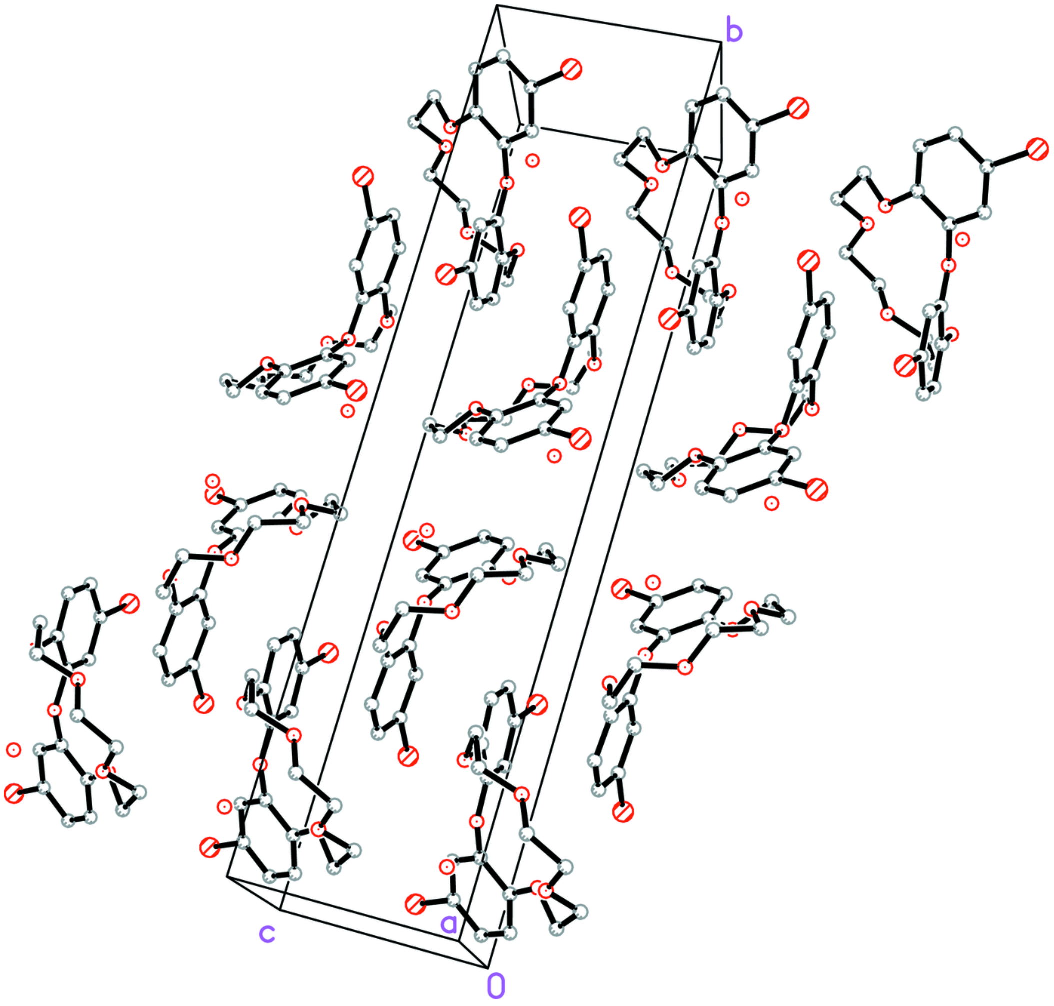
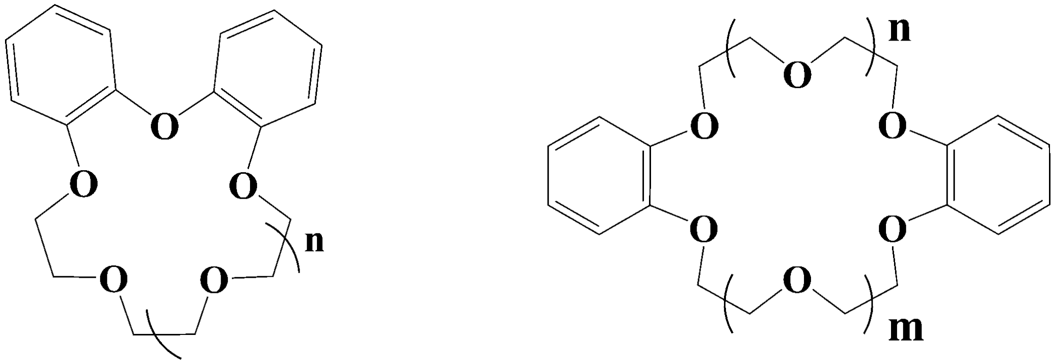


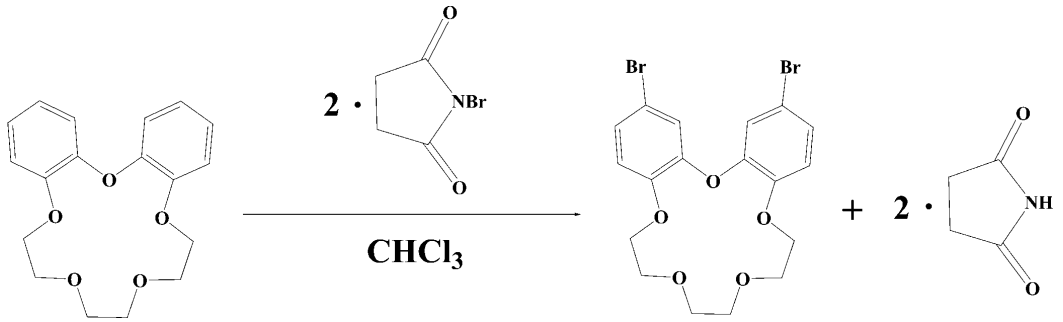
Fig. 3



(I)

(II)

Fig. 4



(III)

(IV)