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2,9,16,19,22,25-Hexaoxatetracyclo-[24.4.0.2^{4,7}.0^{10,15}]dotriacont-1(26),4,6,10(15),11,13,27,29,31-nonaene

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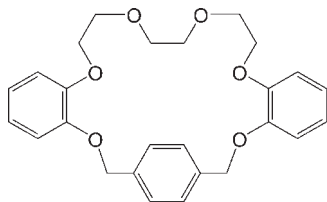
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.116; data-to-parameter ratio = 13.5.

The title 22-crown-6 unit, $\text{C}_{26}\text{H}_{28}\text{O}_6$, comprising of three benzo groups and triethylene glycol, was prepared by the reaction of α,α' -dibromo-*p*-xylene with 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane in the presence of Cs_2CO_3 with tetrahydrofuran (THF) and recrystallized from dichloromethane-hexane (1:20 *v/v*) at room temperature. In the molecular structure, two O atoms of the central ethylene glycol in the triethylene glycol unit exhibit *exo* conformations due to intramolecular C—H \cdots O interactions. A number of C—H \cdots O and C—H \cdots π intermolecular interactions contribute to the stabilization of the crystal packing.

Related literature

For the preparation of related compounds, see: Sim *et al.* (2001); Weber & Vögtle (1976). For a related structure, see: Sim *et al.* (2001). For background to crown ether-based macrocyclic compounds and their inclusion behaviour, see: Gokel & Korzeniowski (1982); Izatt & Christensen (1981); Lindoy (1989); Pedersen (1967); Vögtle & Weber (1985); Weber *et al.* (1989); Wolf *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{26}\text{H}_{28}\text{O}_6$
 $M_r = 436.48$
 Monoclinic, $P2_1/c$
 $a = 12.348$ (3) Å
 $b = 18.908$ (4) Å
 $c = 9.824$ (2) Å
 $\beta = 105.70$ (3)°

 $V = 2208.0$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.25 \times 0.20 \times 0.10$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: none
 4142 measured reflections

 3896 independent reflections
 2654 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.057$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.116$
 $S = 1.02$
 3896 reflections

 289 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C9—H9A \cdots O2	0.97	2.62	3.114 (2)	112
C10—H10A \cdots O5	0.97	2.70	3.117 (2)	106
C2—H2 \cdots O1 ⁱ	0.93	2.76	3.471 (2)	134
C7—H7B \cdots O5 ⁱⁱ	0.97	2.72	3.607 (2)	153
C11—H11B \cdots O1 ⁱⁱ	0.97	2.82	3.501 (3)	128
C11—H11B \cdots O2 ⁱⁱ	0.97	2.90	3.798 (3)	154
C12—H12B \cdots O4 ⁱⁱⁱ	0.97	2.42	3.294 (3)	149
C25—H25 \cdots O3 ⁱⁱ	0.93	2.71	3.433 (2)	135
C12—H12A \cdots Cg1 ⁱⁱ	0.97	2.76	3.47	138
C21—H21 \cdots Cg2 ⁱ	0.93	2.97	3.47	115
C26—H26A \cdots Cg3 ⁱ	0.97	3.06	3.83	138

 Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x, -y, -z + 2$; (iii) $-x, -y, -z + 1$. Cg1, Cg2 and Cg3 are the centroids of the C1–C6, C13–C18 and C20–C25 rings, respectively.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus (Bruker, 2000); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2101).

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supplementary materials

Acta Cryst. (2009). E65, o2369-o2370 [doi:10.1107/S1600536809035399]

2,9,16,19,22,25-Hexaoxatetracyclo[24.4.0.2^{4,7}.0^{10,15}]dotriaconta-1(26),4,6,10(15),11,13,27,29,31-nonaene

J. Y. Lee, J.-E. Lee, W. Sim and K.-M. Park

Comment

An extraordinary variety of crown ether-based macrocyclic compounds have been synthesized and reported since crown ether was first discovered (Pedersen, 1967) because their topological interesting as well as their inclusion behaviour (Gokel & Korzeniowski, 1982; Izatt & Christensen, 1981; Lindoy 1989; Vögtle & Weber, 1985; Weber *et al.* 1989). We have also synthesized and reported the preparation and the solid-state structure of new crown ether (I) bearing three aromatic subunits (Sim *et al.*, 2001). As a part of our continuing interest in the development of new crown compounds, the preparation and crystal structure of new crown ether-based macrocyclic compound (II) containing three benzo units of which ring size is larger than that of the previous reported compound (I) are presented here.

The title compound (II), 2,9,16,19,22,25-hexaoxatetracyclo[24.4.2^{4,7}.0.0^{10,15}]-dotriaconta-1(26),4,6,10(15),11,13,27,29,31-nonaene was prepared by the reaction of α,α' -dibromo-*p*-xylene with 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane in the presence of Cs₂CO₃ with tetrahydrofuran (THF) and recrystallized from dichloromethane/hexane (1:20) at room temperature to give colorless single crystals suitable for X-ray analysis.

In the molecular structure of (II), the torsion angles of C—C—O—C connecting A-to-B rings and A-to-C rings aromatic are 167.0 (2)° and 163.4 (2)°, respectively, which indicate that the A ring is situated *trans* to both the B and C rings, with dihedral angles of 57.04 (8)° between A and B and 44.41 (8)° between A and C. The dihedral angle between B and C rings is 14.2 (1)°. The all O—C—C—O and C—C—O—C torsion angles except two C—C—O—C in the triethylene glycol group exhibit *gauche* conformation. Two exceptional C—C—O—C (C10—C9—O3—C8 and C9—C10—O4—C11) torsion angles are *trans* conformation with the values of -162.5 (2)° and -156.1 (2)°, respectively.

Interestingly, two oxygen atoms of the central ethylene glycol in the triethylene glycol unit exhibit the *exo*-orientations which are very different from those found in the ethylene glycol backbone of (I) (Sim *et al.*, 2001) or common crown ether-based compounds. In general, oxygen atoms of ethylene glycol group in crown ether-based compounds favor *endo*-orientation (Wolf *et al.*, 1987). The *exo*-orientations of two oxygen atoms (O3 and O4) in (II) may be due to the intramolecular C—H \cdots O interactions; 2.62 Å for C9—H9A \cdots O2 and 2.70 Å for C10—H10A \cdots O6 (Fig. 1 & Table 1).

The crystal packing is stabilized by not only intermolecular C—H \cdots O hydrogen bonds with C—H \cdots O separation in the range of 2.71–2.90 Å but intermolecular C—H \cdots π interactions with C—H \cdots C_g separations in the range of 2.76–3.06 Å (Fig. 2 & Table 1; C_g is the centroid of aromatic ring).

Experimental

To a refluxing suspension of caesium carbonate (15.2 mmol) in THF under nitrogen was added dropwise a solution of α,α' -dibromo-*p*-xylene (3.75 mmol) and 1,8-bis(2-hydroxyphenoxy)-3,6-dioxaoctane (3.79 mmol) in THF over a period of 3 h. The mixture was then refluxed for an additional 24 h. After cooling to room temperature, 10% aqueous hydrochloric acid

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was added. The solvent was removed under reduced pressure and the residual mixture was extracted with dichloromethane. The organic layer was washed with water, dried over anhydrous magnesium sulfate, and evaporated *in vacuo*. The crude product was chromatographed on a silica-gel column using a mixed solvent of ethyl acetate and *n*-hexane (1:1) as eluent, and recrystallized from dichloromethane/*n*-hexane (1:20, *v/v*) to give as a crystalline solid in 70% yield (m.p. 424 K). IR (KBr pellet): 2926, 1600, 1504, 1235, 1126, 996 and 735 cm^{-1} . ^1H NMR (CDCl_3): δ 7.54 (d, 4H, Ar-*H*), 7.06–6.91 (m, 8H, Ar-*H*), 5.06 (s, 4H, ArCH₂O), 4.12 (t, 4H, ArOCH₂CH₂OCH₂), 3.88 (t, 4H, ArOCH₂CH₂OCH₂) and 3.70 (t, 4H, ArOCH₂CH₂OCH₂).

Refinement

(type here to add refinement details)

Figures

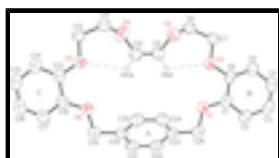


Fig. 1. ORTEP drawing of (II) with the atom-numbering scheme and intramolecular C—H...O interactions (dotted lines). Displacement ellipsoids are drawn at the 50% probability level. All H atoms except two H atoms related to intramolecular C—H...O hydrogen bonds have been omitted for clarity.

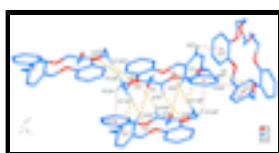


Fig. 2. Intermolecular C—H... π (red dotted lines) and C—H...O (yellow dotted lines) interactions in the title compound. All H atoms except those related to intermolecular interactions have been omitted for clarity. Cg1, Cg2 and Cg3 denote the centroids of rings consisting of C1/C2/C3/C4/C5/C6, C13/C14/C15/C16/C17/C18 and C20/C21/C22/C23/C24/C25, respectively. [Symmetry codes: (i) $x, -y + 1/2, z + 1/2$; (ii) $-x, -y, -z + 2$; (iii) $-x, -y, -z + 1$]

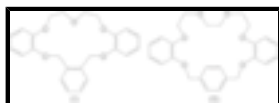


Fig. 3. The structures of (I) and (II).

2,9,16,19,22,25-Hexaoxatetracyclo[24.4.0.2^{4,7}.0^{10,15}]dotriaconta- 1(26),4,6,10 (15),11,13,27,29,31-nonaene

Crystal data

$\text{C}_{26}\text{H}_{28}\text{O}_6$

$M_r = 436.48$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.348$ (3) Å

$b = 18.908$ (4) Å

$c = 9.824$ (2) Å

$\beta = 105.70$ (3)°

$V = 2208.0$ (8) Å³

$Z = 4$

$F_{000} = 928$

$D_x = 1.313$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3896 reflections

$\theta = 1.7$ – 25.0 °

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Plate, colorless

$0.25 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector

2654 reflections with $I > 2\sigma(I)$

diffractometer
 Radiation source: fine-focus sealed tube $R_{\text{int}} = 0.057$
 Monochromator: graphite $\theta_{\text{max}} = 25.1^\circ$
 $T = 293$ K $\theta_{\text{min}} = 1.7^\circ$
 ω scans $h = -14 \rightarrow 14$
 Absorption correction: none $k = 0 \rightarrow 22$
 4142 measured reflections $l = -11 \rightarrow 0$
 3896 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.041$ H-atom parameters constrained
 $wR(F^2) = 0.116$ $w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.0279P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.02$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 3896 reflections $\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$
 289 parameters $\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.14749 (11)	0.20218 (7)	1.10961 (13)	0.0498 (3)
O2	-0.28741 (11)	0.11040 (7)	0.97137 (13)	0.0520 (4)
O3	-0.17603 (11)	0.00949 (7)	0.83350 (15)	0.0568 (4)
O4	-0.00678 (11)	0.00265 (9)	0.68633 (14)	0.0624 (4)
O5	0.23169 (10)	0.03178 (7)	0.73303 (12)	0.0474 (3)
O6	0.29941 (11)	0.14286 (7)	0.87761 (14)	0.0552 (4)
C1	-0.23684 (15)	0.18893 (10)	1.16215 (19)	0.0446 (4)
C2	-0.25554 (17)	0.21956 (11)	1.2812 (2)	0.0538 (5)
H2	-0.2057	0.2534	1.3313	0.065*
C3	-0.34797 (19)	0.20021 (12)	1.3264 (2)	0.0626 (6)

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H3	-0.3593	0.2204	1.4077	0.075*
C4	-0.42279 (19)	0.15159 (12)	1.2525 (2)	0.0639 (6)
H4	-0.4854	0.1393	1.2828	0.077*
C5	-0.40553 (17)	0.12059 (11)	1.1322 (2)	0.0571 (5)
H5	-0.4568	0.0876	1.0820	0.069*
C6	-0.31315 (15)	0.13823 (10)	1.08660 (18)	0.0449 (4)
C7	-0.33649 (16)	0.04425 (10)	0.9193 (2)	0.0509 (5)
H7A	-0.4178	0.0485	0.8889	0.061*
H7B	-0.3169	0.0088	0.9933	0.061*
C8	-0.29280 (16)	0.02303 (12)	0.7974 (2)	0.0557 (5)
H8A	-0.3324	-0.0192	0.7546	0.067*
H8B	-0.3098	0.0603	0.7271	0.067*
C9	-0.11025 (18)	0.06755 (10)	0.8132 (2)	0.0567 (5)
H9A	-0.1033	0.1014	0.8893	0.068*
H9B	-0.1464	0.0910	0.7247	0.068*
C10	0.00358 (16)	0.04204 (11)	0.8106 (2)	0.0532 (5)
H10A	0.0533	0.0820	0.8129	0.064*
H10B	0.0354	0.0127	0.8927	0.064*
C11	0.07916 (16)	-0.04707 (10)	0.6910 (2)	0.0551 (5)
H11A	0.0465	-0.0882	0.6360	0.066*
H11B	0.1099	-0.0623	0.7881	0.066*
C12	0.17270 (15)	-0.02012 (10)	0.6366 (2)	0.0480 (5)
H12A	0.2229	-0.0585	0.6292	0.058*
H12B	0.1430	0.0006	0.5436	0.058*
C13	0.31566 (15)	0.06729 (10)	0.69757 (18)	0.0426 (4)
C14	0.36356 (17)	0.04880 (11)	0.5914 (2)	0.0541 (5)
H14	0.3391	0.0084	0.5380	0.065*
C15	0.44755 (18)	0.08973 (12)	0.5636 (2)	0.0617 (6)
H15	0.4794	0.0768	0.4916	0.074*
C16	0.48412 (16)	0.14911 (12)	0.6415 (2)	0.0588 (6)
H16	0.5405	0.1767	0.6223	0.071*
C17	0.43756 (16)	0.16805 (11)	0.7484 (2)	0.0530 (5)
H17	0.4633	0.2083	0.8018	0.064*
C18	0.35325 (14)	0.12816 (10)	0.77722 (19)	0.0427 (4)
C19	0.30700 (16)	0.21254 (10)	0.9329 (2)	0.0521 (5)
H19A	0.2975	0.2468	0.8571	0.063*
H19B	0.3801	0.2199	0.9994	0.063*
C20	0.21604 (15)	0.22107 (9)	1.00562 (19)	0.0457 (4)
C21	0.16951 (16)	0.28661 (10)	1.0145 (2)	0.0501 (5)
H21	0.1968	0.3260	0.9777	0.060*
C22	0.08275 (17)	0.29458 (10)	1.0775 (2)	0.0503 (5)
H22	0.0532	0.3394	1.0833	0.060*
C23	0.03947 (16)	0.23753 (10)	1.1316 (2)	0.0492 (5)
C24	0.08909 (18)	0.17237 (10)	1.1269 (2)	0.0565 (5)
H24	0.0634	0.1333	1.1664	0.068*
C25	0.17552 (17)	0.16431 (10)	1.0652 (2)	0.0548 (5)
H25	0.2073	0.1198	1.0635	0.066*
C26	-0.05795 (17)	0.24454 (11)	1.1932 (2)	0.0574 (5)
H26A	-0.0373	0.2284	1.2905	0.069*

H26B −0.0813 0.2936 1.1914 0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0493 (7)	0.0561 (8)	0.0464 (7)	−0.0046 (6)	0.0169 (6)	−0.0083 (6)
O2	0.0533 (8)	0.0572 (8)	0.0480 (7)	−0.0055 (6)	0.0178 (6)	−0.0082 (6)
O3	0.0508 (8)	0.0470 (8)	0.0751 (9)	0.0046 (6)	0.0214 (7)	0.0069 (7)
O4	0.0447 (7)	0.0944 (11)	0.0434 (7)	0.0134 (8)	0.0038 (6)	−0.0129 (7)
O5	0.0492 (7)	0.0495 (7)	0.0453 (7)	−0.0086 (6)	0.0156 (6)	−0.0089 (6)
O6	0.0656 (9)	0.0480 (8)	0.0600 (8)	−0.0131 (7)	0.0305 (7)	−0.0144 (6)
C1	0.0464 (10)	0.0450 (11)	0.0425 (10)	0.0107 (8)	0.0121 (9)	0.0034 (8)
C2	0.0593 (12)	0.0511 (12)	0.0526 (12)	0.0106 (10)	0.0179 (10)	−0.0054 (9)
C3	0.0652 (13)	0.0716 (14)	0.0568 (13)	0.0154 (12)	0.0268 (11)	−0.0045 (11)
C4	0.0554 (12)	0.0785 (16)	0.0661 (14)	0.0117 (12)	0.0305 (11)	0.0083 (12)
C5	0.0474 (11)	0.0663 (14)	0.0587 (13)	0.0043 (10)	0.0163 (10)	0.0020 (11)
C6	0.0449 (10)	0.0507 (11)	0.0406 (10)	0.0109 (9)	0.0139 (8)	0.0031 (9)
C7	0.0446 (10)	0.0529 (12)	0.0528 (11)	−0.0026 (9)	0.0092 (9)	−0.0038 (9)
C8	0.0479 (11)	0.0608 (12)	0.0550 (12)	−0.0029 (10)	0.0080 (9)	−0.0118 (10)
C9	0.0617 (13)	0.0452 (11)	0.0686 (13)	0.0005 (10)	0.0269 (11)	0.0013 (10)
C10	0.0517 (11)	0.0608 (13)	0.0468 (11)	−0.0001 (10)	0.0127 (9)	−0.0047 (10)
C11	0.0494 (11)	0.0522 (12)	0.0626 (12)	−0.0069 (10)	0.0129 (10)	−0.0170 (10)
C12	0.0466 (11)	0.0488 (11)	0.0455 (10)	0.0017 (9)	0.0070 (9)	−0.0105 (9)
C13	0.0396 (10)	0.0469 (11)	0.0412 (10)	0.0054 (8)	0.0107 (8)	0.0063 (8)
C14	0.0577 (12)	0.0583 (12)	0.0500 (11)	0.0051 (10)	0.0209 (10)	−0.0012 (10)
C15	0.0609 (13)	0.0730 (15)	0.0609 (13)	0.0107 (12)	0.0328 (11)	0.0073 (12)
C16	0.0469 (11)	0.0640 (14)	0.0714 (14)	0.0053 (10)	0.0261 (11)	0.0164 (12)
C17	0.0442 (10)	0.0521 (11)	0.0617 (13)	−0.0023 (9)	0.0130 (10)	0.0027 (10)
C18	0.0406 (10)	0.0445 (10)	0.0440 (10)	0.0028 (8)	0.0133 (8)	0.0031 (8)
C19	0.0529 (12)	0.0452 (11)	0.0591 (12)	−0.0097 (9)	0.0168 (10)	−0.0106 (9)
C20	0.0462 (10)	0.0429 (10)	0.0452 (10)	−0.0079 (8)	0.0073 (9)	−0.0096 (8)
C21	0.0584 (12)	0.0397 (10)	0.0499 (11)	−0.0091 (9)	0.0106 (9)	−0.0050 (9)
C22	0.0595 (12)	0.0394 (10)	0.0496 (11)	−0.0002 (9)	0.0106 (10)	−0.0082 (9)
C23	0.0516 (11)	0.0464 (11)	0.0488 (11)	−0.0063 (9)	0.0121 (9)	−0.0148 (9)
C24	0.0676 (13)	0.0405 (11)	0.0681 (14)	−0.0068 (10)	0.0296 (11)	−0.0043 (10)
C25	0.0629 (13)	0.0379 (11)	0.0679 (13)	0.0010 (9)	0.0250 (11)	−0.0031 (9)
C26	0.0605 (13)	0.0522 (12)	0.0618 (13)	−0.0069 (10)	0.0205 (11)	−0.0191 (10)

Geometric parameters (Å, °)

O1—C1	1.362 (2)	C11—C12	1.488 (3)
O1—C26	1.431 (2)	C11—H11A	0.9700
O2—C6	1.362 (2)	C11—H11B	0.9700
O2—C7	1.423 (2)	C12—H12A	0.9700
O3—C9	1.412 (2)	C12—H12B	0.9700
O3—C8	1.412 (2)	C13—C14	1.376 (3)
O4—C10	1.406 (2)	C13—C18	1.398 (3)
O4—C11	1.409 (2)	C14—C15	1.379 (3)
O5—C13	1.357 (2)	C14—H14	0.9300

supplementary materials

O5—C12	1.420 (2)	C15—C16	1.365 (3)
O6—C18	1.359 (2)	C15—H15	0.9300
O6—C19	1.418 (2)	C16—C17	1.374 (3)
C1—C2	1.380 (3)	C16—H16	0.9300
C1—C6	1.407 (3)	C17—C18	1.376 (3)
C2—C3	1.381 (3)	C17—H17	0.9300
C2—H2	0.9300	C19—C20	1.494 (3)
C3—C4	1.365 (3)	C19—H19A	0.9700
C3—H3	0.9300	C19—H19B	0.9700
C4—C5	1.386 (3)	C20—C21	1.379 (3)
C4—H4	0.9300	C20—C25	1.380 (3)
C5—C6	1.375 (3)	C21—C22	1.383 (3)
C5—H5	0.9300	C21—H21	0.9300
C7—C8	1.495 (3)	C22—C23	1.373 (3)
C7—H7A	0.9700	C22—H22	0.9300
C7—H7B	0.9700	C23—C24	1.382 (3)
C8—H8A	0.9700	C23—C26	1.491 (3)
C8—H8B	0.9700	C24—C25	1.371 (3)
C9—C10	1.493 (3)	C24—H24	0.9300
C9—H9A	0.9700	C25—H25	0.9300
C9—H9B	0.9700	C26—H26A	0.9700
C10—H10A	0.9700	C26—H26B	0.9700
C10—H10B	0.9700		
C1—O1—C26	117.71 (14)	O5—C12—C11	107.74 (14)
C6—O2—C7	117.76 (14)	O5—C12—H12A	110.2
C9—O3—C8	114.27 (16)	C11—C12—H12A	110.2
C10—O4—C11	115.74 (15)	O5—C12—H12B	110.2
C13—O5—C12	117.39 (13)	C11—C12—H12B	110.2
C18—O6—C19	118.25 (14)	H12A—C12—H12B	108.5
O1—C1—C2	125.65 (18)	O5—C13—C14	125.71 (17)
O1—C1—C6	114.90 (15)	O5—C13—C18	115.11 (15)
C2—C1—C6	119.44 (18)	C14—C13—C18	119.17 (17)
C1—C2—C3	120.2 (2)	C13—C14—C15	120.5 (2)
C1—C2—H2	119.9	C13—C14—H14	119.7
C3—C2—H2	119.9	C15—C14—H14	119.7
C4—C3—C2	120.5 (2)	C16—C15—C14	120.18 (19)
C4—C3—H3	119.8	C16—C15—H15	119.9
C2—C3—H3	119.8	C14—C15—H15	119.9
C3—C4—C5	120.0 (2)	C15—C16—C17	119.98 (19)
C3—C4—H4	120.0	C15—C16—H16	120.0
C5—C4—H4	120.0	C17—C16—H16	120.0
C6—C5—C4	120.5 (2)	C16—C17—C18	120.7 (2)
C6—C5—H5	119.7	C16—C17—H17	119.6
C4—C5—H5	119.7	C18—C17—H17	119.6
O2—C6—C5	125.33 (18)	O6—C18—C17	125.69 (17)
O2—C6—C1	115.35 (16)	O6—C18—C13	114.89 (15)
C5—C6—C1	119.32 (17)	C17—C18—C13	119.41 (17)
O2—C7—C8	108.22 (16)	O6—C19—C20	107.65 (15)
O2—C7—H7A	110.1	O6—C19—H19A	110.2

C8—C7—H7A	110.1	C20—C19—H19A	110.2
O2—C7—H7B	110.1	O6—C19—H19B	110.2
C8—C7—H7B	110.1	C20—C19—H19B	110.2
H7A—C7—H7B	108.4	H19A—C19—H19B	108.5
O3—C8—C7	114.40 (16)	C21—C20—C25	117.85 (18)
O3—C8—H8A	108.7	C21—C20—C19	120.50 (17)
C7—C8—H8A	108.7	C25—C20—C19	121.65 (17)
O3—C8—H8B	108.7	C20—C21—C22	120.86 (18)
C7—C8—H8B	108.7	C20—C21—H21	119.6
H8A—C8—H8B	107.6	C22—C21—H21	119.6
O3—C9—C10	109.36 (16)	C23—C22—C21	121.17 (18)
O3—C9—H9A	109.8	C23—C22—H22	119.4
C10—C9—H9A	109.8	C21—C22—H22	119.4
O3—C9—H9B	109.8	C22—C23—C24	117.69 (18)
C10—C9—H9B	109.8	C22—C23—C26	121.89 (18)
H9A—C9—H9B	108.2	C24—C23—C26	120.41 (18)
O4—C10—C9	108.74 (17)	C25—C24—C23	121.26 (19)
O4—C10—H10A	109.9	C25—C24—H24	119.4
C9—C10—H10A	109.9	C23—C24—H24	119.4
O4—C10—H10B	109.9	C24—C25—C20	121.07 (19)
C9—C10—H10B	109.9	C24—C25—H25	119.5
H10A—C10—H10B	108.3	C20—C25—H25	119.5
O4—C11—C12	114.25 (17)	O1—C26—C23	107.49 (15)
O4—C11—H11A	108.7	O1—C26—H26A	110.2
C12—C11—H11A	108.7	C23—C26—H26A	110.2
O4—C11—H11B	108.7	O1—C26—H26B	110.2
C12—C11—H11B	108.7	C23—C26—H26B	110.2
H11A—C11—H11B	107.6	H26A—C26—H26B	108.5
C26—O1—C1—C2	8.2 (3)	C13—C14—C15—C16	0.1 (3)
C26—O1—C1—C6	-170.39 (16)	C14—C15—C16—C17	0.3 (3)
O1—C1—C2—C3	-178.12 (17)	C15—C16—C17—C18	-0.7 (3)
C6—C1—C2—C3	0.5 (3)	C19—O6—C18—C17	17.7 (3)
C1—C2—C3—C4	-1.2 (3)	C19—O6—C18—C13	-161.32 (17)
C2—C3—C4—C5	0.8 (3)	C16—C17—C18—O6	-178.25 (18)
C3—C4—C5—C6	0.2 (3)	C16—C17—C18—C13	0.7 (3)
C7—O2—C6—C5	-20.6 (3)	O5—C13—C18—O6	-0.4 (2)
C7—O2—C6—C1	159.34 (16)	C14—C13—C18—O6	178.72 (16)
C4—C5—C6—O2	179.09 (18)	O5—C13—C18—C17	-179.49 (16)
C4—C5—C6—C1	-0.9 (3)	C14—C13—C18—C17	-0.3 (3)
O1—C1—C6—O2	-0.7 (2)	C18—O6—C19—C20	163.38 (15)
C2—C1—C6—O2	-179.42 (16)	O6—C19—C20—C21	-151.26 (17)
O1—C1—C6—C5	179.28 (16)	O6—C19—C20—C25	28.5 (2)
C2—C1—C6—C5	0.5 (3)	C25—C20—C21—C22	-1.9 (3)
C6—O2—C7—C8	-177.71 (15)	C19—C20—C21—C22	177.84 (17)
C9—O3—C8—C7	-95.6 (2)	C20—C21—C22—C23	-0.7 (3)
O2—C7—C8—O3	64.9 (2)	C21—C22—C23—C24	3.0 (3)
C8—O3—C9—C10	-162.52 (16)	C21—C22—C23—C26	-176.78 (18)
C11—O4—C10—C9	-156.07 (16)	C22—C23—C24—C25	-2.6 (3)
O3—C9—C10—O4	69.2 (2)	C26—C23—C24—C25	177.11 (19)

supplementary materials

C10—O4—C11—C12	-93.9 (2)	C23—C24—C25—C20	0.0 (3)
C13—O5—C12—C11	-175.06 (15)	C21—C20—C25—C24	2.2 (3)
O4—C11—C12—O5	68.5 (2)	C19—C20—C25—C24	-177.50 (19)
C12—O5—C13—C14	-13.4 (3)	C1—O1—C26—C23	166.99 (15)
C12—O5—C13—C18	165.73 (15)	C22—C23—C26—O1	118.0 (2)
O5—C13—C14—C15	179.01 (17)	C24—C23—C26—O1	-61.7 (2)
C18—C13—C14—C15	-0.1 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C9—H9A \cdots O2	0.97	2.62	3.114 (2)	112
C10—H10A \cdots O5	0.97	2.70	3.117 (2)	106
C2—H2 \cdots O1 ⁱ	0.93	2.76	3.471 (2)	134
C7—H7B \cdots O5 ⁱⁱ	0.97	2.72	3.607 (2)	153
C11—H11B \cdots O1 ⁱⁱ	0.97	2.82	3.501 (3)	128
C11—H11B \cdots O2 ⁱⁱ	0.97	2.90	3.798 (3)	154
C12—H12B \cdots O4 ⁱⁱⁱ	0.97	2.42	3.294 (3)	149
C25—H25 \cdots O3 ⁱⁱ	0.93	2.71	3.433 (2)	135
C12—H12A \cdots Cg1 ⁱⁱ	0.97	2.76	3.47	138
C21—H21 \cdots Cg2 ⁱ	0.93	2.97	3.47	115
C26—H26A \cdots Cg3 ⁱ	0.97	3.06	3.83	138

Symmetry codes: (i) $x, -y+1/2, z+1/2$; (ii) $-x, -y, -z+2$; (iii) $-x, -y, -z+1$.

Fig. 1

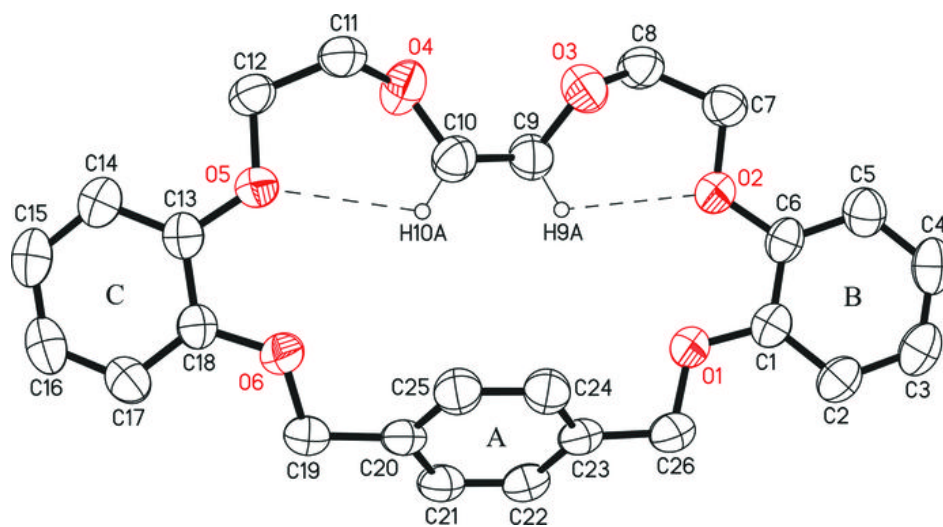


Fig. 2

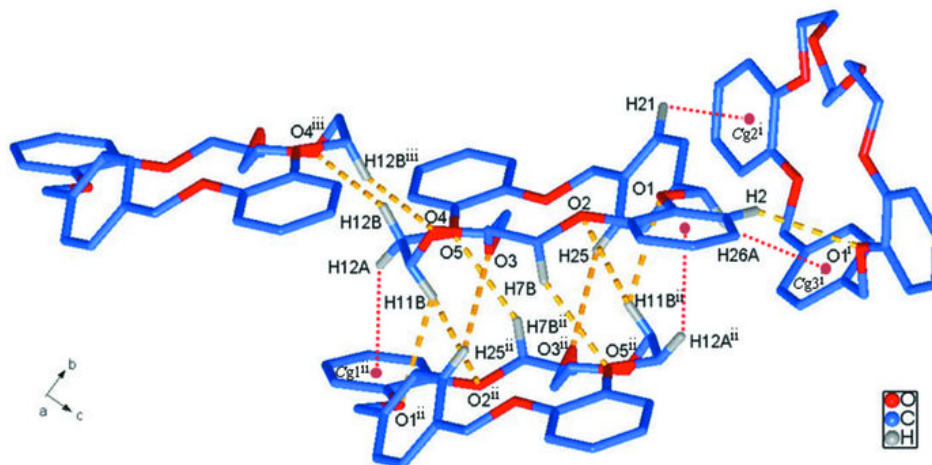


Fig. 3

