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Redetermination of 1,13-diphenyl-2,4,6,8,10,12-hexaoxatridecane at 161 K

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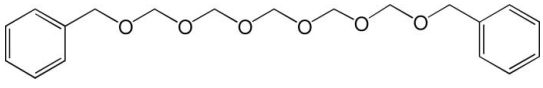
Key indicators: single-crystal X-ray study; T = 161 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.082; data-to-parameter ratio = 15.0.

The title compound, $C_{19}H_{24}O_6$, crystallizes with two half-molecules per asymmetric unit; each molecule has a crystallographic twofold axis passing through the central CH_2 group. The two molecules have different orientations of the terminal benzyl groups. The C—O bond lengths in the central section of each polyoxymethylene helix are almost constant. The average C—O bond length, corrected for librational motion, is 1.421 Å. The molecules are connected into layers by intermolecular C—H···O and C—H···π(phenyl) interactions. The structure was previously reported by Noe, Miculka & Bats [(1994), *Angew. Chem. Int. Ed. Engl.* **33**, 1476–1478].

Related literature

The crystal structure of the title compound, determined at room temperature, was previously reported by Noe *et al.* (1994). An isomorphous crystal structure has been reported by Bats *et al.* (2007) and the structure of a closely related molecule by Bats *et al.* (2001). For the libration correction, see Farrugia (1999).



Experimental

Crystal data

$C_{19}H_{24}O_6$
M_r = 348.38
Monoclinic, $C2$
a = 40.733 (9) Å
b = 5.4310 (10) Å
β = 97.34 (2)°
V = 1813.6 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.78 mm^{-1}
T = 161 (2) K
0.55 × 0.50 × 0.10 mm

Data collection

Enraf–Nonius CAD4 diffractometer
Absorption correction: numerical using eight faces (*SHELXTL*; Sheldrick, 1996)
T_{min} = 0.690, T_{max} = 0.933
3681 measured reflections
3429 independent reflections
3311 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
3 standard reflections
frequency: 92 min
intensity decay: <1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.10
3429 reflections
228 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Flack (1983), with 1519 Friedel pairs
Flack parameter: 0.23 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4	0.95	2.44	3.333 (2)	158
C8—H8B···O6	0.99	2.60	3.511 (2)	153
C10—H10···O5^{i}	0.99	2.58	3.477 (2)	150
C18—H18A···O2^{ii}	0.99	2.60	3.541 (2)	160
C19—H19A···O1^{iii}	0.99	2.65	3.520 (2)	147
C19—H19B···O3^{iv}	0.99	2.64	3.542 (2)	152
C3—H3···CgB	0.95	2.92	3.715	143
C6—H6···CgB^{v}	0.95	3.00	3.745	136
C13—H13···CgA^{iii}	0.95	3.07	3.704	126
C16—H16···CgA^{ii}	0.95	3.19	3.913	134

Symmetry codes: (i) $-x+1, y, -z+1$; (ii) $x, y, z+1$; (iii) $x, y-1, z$; (iv) $-x+1, y-1, -z+1$; (v) $x, y+1, z-1$. CgA and CgB are the centroids of the phenyl rings of molecules A and B, respectively.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2020).

References

Bats, J. W., Kruse, J. & Noe, C. R. (2001). Private communication (refcode QULVOI). CCDC, 12 Union Road, Cambridge, England.
Bats, J. W., Miculka, C. & Noe, C. R. (2007). *Acta Cryst.* C**63**, o190–o192.
Enraf–Nonius. (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Noe, C. R., Miculka, C. & Bats, J. W. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 1476–1478.
Sheldrick, G. M. (1996). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

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supplementary materials

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Redetermination of 1,13-diphenyl-2,4,6,8,10,12-hexaoxatridecane at 161 K

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Comment

The crystal structure of (I) was previously determined at room temperature (Noe *et al.*, 1994). A small but significant alternation of the C—O bond lengths was reported, which was not understood at that time. To clearify this point a redetermination of (I) at low temperature has now been undertaken. Crystals of (I) undergo a reversible phase transition at approximately 140 K, accompanied by a splitting of the reflection profiles in the low temperature phase. The measurements of (I) were performed at 161 K, which is well above the phase transition temperature.

The structure of (I) is isomorphous with the crystal structure of 1,17-diphenyl-2,4,6,8,10,12,14,16-octaoxaheptadecane (Bats *et al.*, 2007). The compound has two crystallographically independent molecules (Fig. 1), each displaying crystallographic twofold symmetry with the axis passing through the central CH_2 group. The two independent molecules have different orientations of the terminal benzyl groups. The phenyl group of molecule A is synperiplanar with the C7—O1 bond. The phenyl group of molecule B is almost perpendicular to the C17—O4 bond.

In the roomtemperature determination of (I) we observed a systematic C—O bond length variation. This effect is not observed in the present low temperature structure determination. Reprocessing of the room temperature data of (I) showed this bond length variation to result from using an inappropriate weighting scheme in a polar space group.

The C—O bond lengths in the central section of each helix is almost constant. An average C—O bond length of 1.416 Å is observed in the regions C8—C8($1-x,y,-z$) and C18—C18($1-x,y,1-z$). The polyoxymethylene helices (without the benzyl groups) behave as rigid bodies with rather large librational motion along the helix axis [46 (3)$^{\circ 2}$ for molecule A and 41 (3)$^{\circ 2}$ for molecule B], but with almost no librational motion about axes perpendicular to the molecular axis. The average C—O bond length, corrected for librational motion, is 1.421 Å. Values of 1.420 Å and 1.419 Å have been observed in the structures of the related compounds 1,15-diphenyl-heptaoxapentadecane (Bats *et al.*, 2001) and 1,13-diphenyl-hexaoxaheptadecane (Bats *et al.*, 2007).

The C—O—C bond angles in (I) range between 113.74 (10)° and 114.45 (8)° and are almost constant with an average value of 114.20°. The O—C—O angles range between 112.23 (12)° and 113.14 (11)° with a average values of 112.58°. The C—O—C—O torsion angles vary between 60.80 (14)° and 68.74 (14)° with an average value of 65.74°. Almost constant torsion angles, corresponding to an undisturbed helix, are found in molecule A. The helix of molecule B is slightly bend, resulting in deviations of the C—O—C—O torsion angles by up to 4° from their average value.

The crystal packing of (I) is stabilized by a number of intermolecular C—H···O and C—H···π(phenyl) interactions. It is similar to the crystal packing of the isomorphous compound reported by Bats *et al.* (2007).

Experimental

Compound (I) was prepared as described by Noe *et al.* (1994). Thin plates were obtained by crystallization from chloroform-hexane (1:1) at low temperature.

Refinement

The H atoms were located in a difference Fourier map and were refined as riding with C(sp^2)—H = 0.95 Å, $C_{secondary}$—H = 0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C). Friedel opposites were not merged. The absolute structure was determined from the anomalous scattering contribution of the O atoms, using 1519 Friedel pairs. The thermal motion analysis was performed with the *WinGX* program package (Farrugia, 1999).

Figures

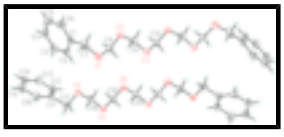


Fig. 1. The structures of the two independent molecules of (I), with the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level. Molecule A is at the top and molecule B at the bottom. Unlabelled atoms are related to labelled atoms by the symmetry operator (1 − *x*, *y*, −*z*) in molecule A and by (1 − *x*, *y*, 1 − *z*) in molecule B.

1,13-diphenyl-2,4,6,8,10,12-hexaoxatridecane

Crystal data

$C_{19}H_{24}O_6$	F_{000} = 744
M_r = 348.38	D_x = 1.276 Mg m^{-3}
Monoclinic, *C*2	Cu *K*α radiation λ = 1.54180 Å
Hall symbol: C 2y	Cell parameters from 25 reflections
a = 40.733 (9) Å	θ = 39–61º
b = 5.4310 (10) Å	μ = 0.78 mm^{-1}
c = 8.266 (3) Å	*T* = 161 (2) K
β = 97.34 (2)º	Plate, colourless
V = 1813.6 (8) Å^3	0.55 × 0.50 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD4 diffractometer	R_{int} = 0.028
Radiation source: fine-focus sealed tube	θ_{max} = 70.0º
Monochromator: graphite	θ_{min} = 2.2º
T = 161(2) K	*h* = −49→48
ω scans	*k* = −6→6
Absorption correction: numerical using eight faces (SHELXTL; Sheldrick, 1996)	*l* = 0→10

T_{min} = 0.690, T_{max} = 0.933	3 standard reflections
3681 measured reflections	every 92 min
3429 independent reflections	intensity decay: <1%
3311 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.46P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.082$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.10$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
3429 reflections	$\Delta\rho_{min} = -0.12$ e Å^{-3}
228 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.00125 (12)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 1519 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.23 (15)

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.41078 (2)	0.51161 (19)	0.28403 (11)	0.0377 (2)
O2	0.43216 (2)	0.4939 (2)	0.03376 (10)	0.0345 (2)
O3	0.48919 (2)	0.5043 (2)	0.12547 (10)	0.0335 (2)
O4	0.39454 (2)	0.04835 (18)	0.62151 (10)	0.0344 (2)
O5	0.45158 (2)	0.01186 (19)	0.70716 (10)	0.0338 (2)
O6	0.47152 (2)	−0.0039 (2)	0.45254 (10)	0.0346 (2)
C1	0.35059 (3)	0.4797 (3)	0.20153 (15)	0.0334 (3)
C2	0.34789 (3)	0.2676 (3)	0.29233 (16)	0.0328 (3)
H2	0.3660	0.2173	0.3689	0.039*
C3	0.31920 (3)	0.1275 (3)	0.27349 (19)	0.0439 (4)
H3	0.3179	−0.0174	0.3367	0.053*

C4	0.29282 (4)	0.1978 (4)	0.1640 (2)	0.0590 (5)
H4	0.2731	0.1024	0.1511	0.071*
C5	0.29506 (4)	0.4070 (4)	0.0732 (2)	0.0658 (6)
H5	0.2767	0.4564	−0.0023	0.079*
C6	0.32371 (4)	0.5474 (4)	0.08976 (18)	0.0513 (4)
H6	0.3250	0.6903	0.0245	0.062*
C7	0.38097 (4)	0.6406 (3)	0.2291 (2)	0.0441 (4)
H7A	0.3837	0.7256	0.1257	0.053*
H7B	0.3774	0.7683	0.3105	0.053*
C8	0.42210 (3)	0.3591 (3)	0.16613 (16)	0.0337 (3)
H8A	0.4042	0.2436	0.1239	0.040*
H8B	0.4410	0.2604	0.2179	0.040*
C9	0.46032 (3)	0.6437 (3)	0.07662 (16)	0.0347 (3)
H9A	0.4640	0.7473	−0.0180	0.042*
H9B	0.4562	0.7547	0.1668	0.042*
C10	0.5000	0.3592 (4)	0.0000	0.0323 (4)
H10	0.5184	0.2519	0.0475	0.039*
C11	0.34710 (3)	−0.1303 (3)	0.71490 (17)	0.0360 (3)
C12	0.33846 (4)	−0.3416 (3)	0.62612 (18)	0.0411 (3)
H12	0.3553	−0.4490	0.5981	0.049*
C13	0.30536 (4)	−0.3979 (3)	0.57764 (19)	0.0445 (4)
H13	0.2996	−0.5416	0.5148	0.053*
C14	0.28097 (4)	−0.2455 (3)	0.6206 (2)	0.0446 (4)
H14	0.2584	−0.2843	0.5875	0.054*
C15	0.28913 (4)	−0.0370 (3)	0.71130 (19)	0.0463 (4)
H15	0.2722	0.0662	0.7426	0.056*
C16	0.32216 (4)	0.0226 (3)	0.75710 (18)	0.0409 (3)
H16	0.3278	0.1687	0.8176	0.049*
C17	0.38279 (4)	−0.0641 (3)	0.76078 (17)	0.0443 (4)
H17A	0.3851	0.0520	0.8539	0.053*
H17B	0.3959	−0.2136	0.7934	0.053*
C18	0.42479 (3)	0.1719 (3)	0.65835 (16)	0.0341 (3)
H18A	0.4229	0.2918	0.7469	0.041*
H18B	0.4295	0.2653	0.5611	0.041*
C19	0.46035 (3)	−0.1411 (3)	0.58093 (17)	0.0351 (3)
H19A	0.4409	−0.2407	0.5366	0.042*
H19B	0.4781	−0.2557	0.6266	0.042*
C20	0.5000	0.1404 (4)	0.5000	0.0335 (4)
H20	0.4959	0.2478	0.5921	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0374 (5)	0.0430 (5)	0.0336 (5)	−0.0069 (5)	0.0086 (4)	−0.0066 (5)
O2	0.0294 (4)	0.0461 (6)	0.0278 (4)	0.0032 (4)	0.0029 (3)	−0.0015 (4)
O3	0.0292 (4)	0.0439 (5)	0.0270 (4)	−0.0025 (4)	0.0019 (3)	−0.0004 (4)
O4	0.0287 (4)	0.0461 (6)	0.0280 (4)	−0.0014 (4)	0.0025 (3)	0.0040 (4)
O5	0.0301 (4)	0.0414 (5)	0.0286 (4)	−0.0020 (4)	−0.0015 (3)	0.0011 (4)

O6	0.0320 (5)	0.0424 (5)	0.0285 (4)	0.0012 (4)	0.0000 (3)	−0.0009 (4)
C1	0.0348 (6)	0.0359 (7)	0.0312 (6)	0.0089 (6)	0.0110 (5)	−0.0002 (6)
C2	0.0293 (6)	0.0344 (7)	0.0346 (7)	0.0019 (5)	0.0036 (5)	−0.0003 (6)
C3	0.0391 (7)	0.0485 (8)	0.0461 (8)	−0.0071 (7)	0.0131 (6)	−0.0122 (7)
C4	0.0305 (8)	0.0827 (14)	0.0630 (11)	−0.0010 (8)	0.0033 (7)	−0.0340 (11)
C5	0.0425 (9)	0.1012 (17)	0.0484 (10)	0.0295 (10)	−0.0145 (7)	−0.0238 (10)
C6	0.0613 (10)	0.0583 (10)	0.0336 (7)	0.0280 (8)	0.0039 (7)	0.0018 (7)
C7	0.0484 (8)	0.0314 (7)	0.0570 (9)	0.0029 (7)	0.0238 (7)	0.0013 (7)
C8	0.0290 (6)	0.0370 (7)	0.0349 (7)	0.0005 (6)	0.0040 (5)	−0.0006 (6)
C9	0.0349 (7)	0.0350 (7)	0.0351 (7)	0.0010 (6)	0.0083 (5)	0.0014 (6)
C10	0.0275 (8)	0.0358 (10)	0.0330 (9)	0.000	0.0017 (7)	0.000
C11	0.0363 (7)	0.0425 (7)	0.0304 (7)	−0.0006 (6)	0.0088 (5)	0.0070 (6)
C12	0.0398 (7)	0.0411 (8)	0.0441 (8)	0.0051 (7)	0.0114 (6)	−0.0011 (7)
C13	0.0477 (8)	0.0374 (8)	0.0492 (8)	−0.0047 (6)	0.0095 (6)	−0.0039 (7)
C14	0.0359 (7)	0.0483 (8)	0.0511 (9)	−0.0052 (7)	0.0108 (6)	0.0035 (7)
C15	0.0418 (8)	0.0453 (9)	0.0541 (9)	0.0085 (7)	0.0145 (7)	−0.0002 (7)
C16	0.0466 (8)	0.0345 (7)	0.0426 (7)	−0.0004 (7)	0.0092 (6)	−0.0019 (6)
C17	0.0417 (8)	0.0610 (10)	0.0299 (7)	−0.0068 (7)	0.0029 (6)	0.0093 (7)
C18	0.0328 (6)	0.0351 (7)	0.0343 (7)	0.0007 (6)	0.0041 (5)	0.0005 (6)
C19	0.0310 (6)	0.0357 (7)	0.0377 (7)	−0.0011 (5)	0.0012 (5)	−0.0002 (6)
C20	0.0332 (9)	0.0321 (9)	0.0350 (9)	0.000	0.0033 (7)	0.000

Geometric parameters (Å, °)

O1—C8	1.4013 (16)	C8—H8B	0.9900
O1—C7	1.4247 (18)	C9—H9A	0.9900
O2—C9	1.4141 (16)	C9—H9B	0.9900
O2—C8	1.4198 (16)	C10—O3^{i}	1.4165 (15)
O3—C9	1.4136 (16)	C10—H10	0.9900
O3—C10	1.4167 (15)	C10—H10^{i}	0.9900
O4—C18	1.4018 (16)	C11—C12	1.383 (2)
O4—C17	1.4380 (16)	C11—C16	1.391 (2)
O5—C18	1.4130 (16)	C11—C17	1.498 (2)
O5—C19	1.4149 (17)	C12—C13	1.391 (2)
O6—C20	1.4135 (15)	C12—H12	0.9500
O6—C19	1.4184 (17)	C13—C14	1.374 (2)
C1—C2	1.387 (2)	C13—H13	0.9500
C1—C6	1.389 (2)	C14—C15	1.375 (2)
C1—C7	1.509 (2)	C14—H14	0.9500
C2—C3	1.3865 (19)	C15—C16	1.389 (2)
C2—H2	0.9500	C15—H15	0.9500
C3—C4	1.367 (2)	C16—H16	0.9500
C3—H3	0.9500	C17—H17A	0.9900
C4—C5	1.371 (3)	C17—H17B	0.9900
C4—H4	0.9500	C18—H18A	0.9900
C5—C6	1.386 (3)	C18—H18B	0.9900
C5—H5	0.9500	C19—H19A	0.9900
C6—H6	0.9500	C19—H19B	0.9900

C7—H7A	0.9900	C20—O6^{ii}	1.4135 (15)
C7—H7B	0.9900	C20—H20	0.9900
C8—H8A	0.9900	C20—H20^{ii}	0.9900
C8—O1—C7	114.31 (10)	O3^{i}—C10—H10^{i}	109.1
C9—O2—C8	114.11 (9)	O3—C10—H10^{i}	109.1
C9—O3—C10	114.45 (8)	H10—C10—H10^{i}	107.9
C18—O4—C17	113.74 (10)	C12—C11—C16	118.94 (13)
C18—O5—C19	114.16 (10)	C12—C11—C17	120.43 (14)
C20—O6—C19	114.45 (8)	C16—C11—C17	120.61 (14)
C2—C1—C6	118.02 (14)	C11—C12—C13	120.46 (14)
C2—C1—C7	121.31 (12)	C11—C12—H12	119.8
C6—C1—C7	120.58 (14)	C13—C12—H12	119.8
C3—C2—C1	121.25 (13)	C14—C13—C12	119.96 (15)
C3—C2—H2	119.4	C14—C13—H13	120.0
C1—C2—H2	119.4	C12—C13—H13	120.0
C4—C3—C2	120.07 (17)	C13—C14—C15	120.30 (14)
C4—C3—H3	120.0	C13—C14—H14	119.8
C2—C3—H3	120.0	C15—C14—H14	119.8
C3—C4—C5	119.47 (17)	C14—C15—C16	119.93 (14)
C3—C4—H4	120.3	C14—C15—H15	120.0
C5—C4—H4	120.3	C16—C15—H15	120.0
C4—C5—C6	121.05 (15)	C15—C16—C11	120.38 (14)
C4—C5—H5	119.5	C15—C16—H16	119.8
C6—C5—H5	119.5	C11—C16—H16	119.8
C5—C6—C1	120.14 (16)	O4—C17—C11	108.10 (11)
C5—C6—H6	119.9	O4—C17—H17A	110.1
C1—C6—H6	119.9	C11—C17—H17A	110.1
O1—C7—C1	114.37 (12)	O4—C17—H17B	110.1
O1—C7—H7A	108.7	C11—C17—H17B	110.1
C1—C7—H7A	108.7	H17A—C17—H17B	108.4
O1—C7—H7B	108.7	O4—C18—O5	113.14 (11)
C1—C7—H7B	108.7	O4—C18—H18A	109.0
H7A—C7—H7B	107.6	O5—C18—H18A	109.0
O1—C8—O2	112.58 (12)	O4—C18—H18B	109.0
O1—C8—H8A	109.1	O5—C18—H18B	109.0
O2—C8—H8A	109.1	H18A—C18—H18B	107.8
O1—C8—H8B	109.1	O5—C19—O6	112.23 (12)
O2—C8—H8B	109.1	O5—C19—H19A	109.2
H8A—C8—H8B	107.8	O6—C19—H19A	109.2
O3—C9—O2	112.45 (12)	O5—C19—H19B	109.2
O3—C9—H9A	109.1	O6—C19—H19B	109.2
O2—C9—H9A	109.1	H19A—C19—H19B	107.9
O3—C9—H9B	109.1	O6—C20—O6^{ii}	112.66 (16)
O2—C9—H9B	109.1	O6—C20—H20	109.1
H9A—C9—H9B	107.8	O6^{ii}—C20—H20	109.1
O3^{i}—C10—O3	112.41 (16)	O6—C20—H20^{ii}	109.1
O3^{i}—C10—H10	109.1	O6^{ii}—C20—H20^{ii}	109.1

O3—C10—H10	109.1	H20—C20—H20^{ii}	107.8
C6—C1—C2—C3	−0.5 (2)	C16—C11—C12—C13	1.0 (2)
C7—C1—C2—C3	176.24 (13)	C17—C11—C12—C13	−177.30 (14)
C1—C2—C3—C4	−0.1 (2)	C11—C12—C13—C14	−1.2 (2)
C2—C3—C4—C5	0.2 (2)	C12—C13—C14—C15	0.1 (2)
C3—C4—C5—C6	0.4 (3)	C13—C14—C15—C16	1.2 (2)
C4—C5—C6—C1	−1.0 (2)	C14—C15—C16—C11	−1.4 (2)
C2—C1—C6—C5	1.0 (2)	C12—C11—C16—C15	0.3 (2)
C7—C1—C6—C5	−175.69 (14)	C17—C11—C16—C15	178.64 (13)
C8—O1—C7—C1	70.25 (15)	C18—O4—C17—C11	165.43 (12)
C2—C1—C7—O1	30.25 (18)	C12—C11—C17—O4	80.05 (17)
C6—C1—C7—O1	−153.13 (13)	C16—C11—C17—O4	−98.27 (16)
C7—O1—C8—O2	66.21 (14)	C17—O4—C18—O5	67.37 (14)
C9—O2—C8—O1	66.01 (14)	C19—O5—C18—O4	68.74 (14)
C10—O3—C9—O2	65.90 (13)	C18—O5—C19—O6	63.10 (13)
C8—O2—C9—O3	66.15 (13)	C20—O6—C19—O5	60.80 (14)
C9—O3—C10—O3^{i}	66.17 (9)	C19—O6—C20—O6^{ii}	66.99 (9)

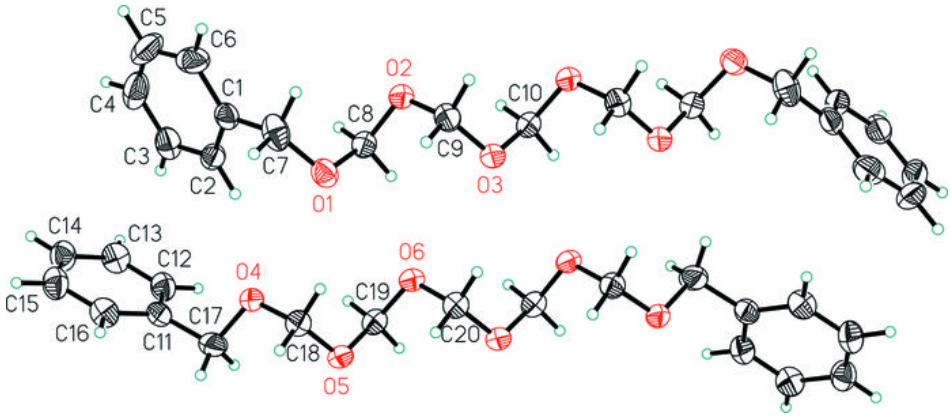
Symmetry codes: (i) −*x*+1, *y*, −*z*; (ii) −*x*+1, *y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4	0.95	2.44	3.333 (2)	158
C8—H8B···O6	0.99	2.60	3.511 (2)	153
C10—H10···O5^{ii}	0.99	2.58	3.477 (2)	150
C18—H18A···O2^{iii}	0.99	2.60	3.541 (2)	160
C19—H19A···O1^{iv}	0.99	2.65	3.520 (2)	147
C19—H19B···O3^{v}	0.99	2.64	3.542 (2)	152
C3—H3···CgB	0.95	2.92	3.715	143
C6—H6···CgB^{vi}	0.95	3.00	3.745	136
C13—H13···CgA^{iv}	0.95	3.07	3.704	126
C16—H16···CgA^{iii}	0.95	3.19	3.913	134

Symmetry codes: (ii) −*x*+1, *y*, −*z*+1; (iii) *x*, *y*, *z*+1; (iv) *x*, *y*−1, *z*; (v) −*x*+1, *y*−1, −*z*+1; (vi) *x*, *y*+1, *z*−1.

Fig. 1


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