Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3′-Dimethoxy-4,4′-(propane-1,3-diyldioxy)dibenzoic acid

Rui-Xue Deng,[a] Pu Liu[b]* and Wei-Yi Zhou[c]

[a]Department of Chemistry, Tianjin University, Tianjin 300072, People's Republic of China, [b]Henan University of Science and Technology, Chemical Engineering and Pharmaceutics College, Henan 471003, People's Republic of China, and [c]Analysis Center, Tianjin University, Tianjin 300072, People's Republic of China
Correspondence e-mail: dengliu20022002@yahoo.com.cn

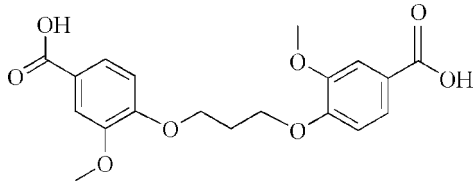
Received 23 September 2007; accepted 26 September 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.135; data-to-parameter ratio = 14.3.

In the title compound, $C_{19}H_{20}O_8$, the two benzene rings are approximately perpendicular to each other [dihedral angle = 83.7 (3)°]. The methoxy and carboxyl groups do not deviate from the planes of the respective benzene rings. Intermolecular O—H···O hydrogen bonds stabilize the crystal structure.

Related literature

For related literature, see: Thurston *et al.* (1996).



Experimental

Crystal data

$C_{19}H_{20}O_8$
M_r = 376.35
Triclinic, $P\overline{1}$
a = 4.8729 (11) Å
b = 7.9736 (18) Å
c = 23.604 (6) Å
α = 93.744 (4)°
β = 94.260 (4)°
γ = 104.173 (4)°
V = 883.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.16 × 0.12 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.982, T_{max} = 0.991
5097 measured reflections
3537 independent reflections
1907 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.135
S = 0.99
3537 reflections
248 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.81	2.618 (3)	166
O7—H7···O8^{ii}	0.82	1.84	2.658 (3)	174

Symmetry codes: (i) $-x+3, -y+2, -z+1$; (ii) $-x-2, -y+3, -z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2410).

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97*. University of Göttingen, Germany.
Thurston, D. E., Bose, D. S., Thompson, A. S., Howard, P. W., Leoni, A., Croker, S. J., Jenkins, T. C., Neidle, S., Hartley, J. A. & Hurley, L. H. (1996). *J. Org. Chem.* **61**, 8141–8147.

supplementary materials

Acta Cryst. (2007). E**63**, o4193 [**doi:10.1107/S1600536807047216**]

3,3'-Dimethoxy-4,4'-(propane-1,3-diyldioxy)dibenzoic acid

R.-X. Deng, P. Liu and W.-Y. Zhou

Comment

The oxygen atoms of carboxyl group can coordinate to metal ions forming various complexes with novel structures. In this paper we synthesize a special flexible ligand containing double aromatic acid connected by propyl spacer. Under nitrogen atmosphere we first made the title compound (I). The single crystals of (I) were obtained and its molecular and crystal structures were characterized by single-crystal X-ray diffraction.

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The dihedral angle between the two benzene ring is 96.3 (3)°, indicating that the two benzene rings are approximately perpendicular to each other. The two methoxy groups are nearly planar with their benzene rings (C2–C7) and (C12–C17) with the r.m.s deviations of 0.0397 (3) and 0.255 (3) Å, respectively. Similarly, two r.m.s deviations of the two carbonyl groups from these two benzene rings are 0.0111 and 0.07233) Å, respectively. The intermolecular O—H···O hydrogen-bonded between the hydroxyl and carbonyl groups form a centrosymmetric dimer (Table 2) and stabilize the crystal structure.

Experimental

The title compound (I) was synthesized according to the literature procedure (Thurston *et al.*, 1996). A mixture of 200 mg of the title compound and 10 ml water was stirred for 1 h and then heated to 413 K for 2 d. Single crystals of (I) suitable for X-ray analysis were obtained after they are cooled to room temperature at 10 ^{o}C/h.

Single crystals of (I) suitable for X-ray analysis were obtained by recrystallized under autogenous pressure.

Refinement

All H atoms were positioned geometrically and refined as riding [C—H = 0.93–0.97 Å, O—H = 0.82 Å]. For CH and CH_2 groups, U_{iso}(H) values were set equal to 1.2U_{eq}(carrier atom) and for the methyl and hydroxy groups they were set equal to 1.5U_{eq}(carrier atom).

Figures

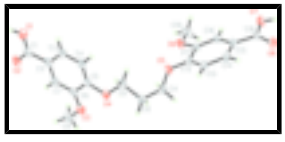


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

3,3'-Dimethoxy-4,4'-(propane-1,3-diyldioxy)dibenzoic acid

Crystal data

$C_{19}H_{20}O_8$	$Z = 2$
$M_r = 376.35$	$F_{000} = 396$
Triclinic, $P\overline{1}$	$D_x = 1.415$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 512 K
$a = 4.8729$ (11) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 7.9736$ (18) Å	Cell parameters from 1045 reflections
$c = 23.604$ (6) Å	$\theta = 2.6$–26.0º
$\alpha = 93.744$ (4)º	$\mu = 0.11$ mm^{-1}
$\beta = 94.260$ (4)º	$T = 294$ (2) K
$\gamma = 104.173$ (4)º	Plate, colourless
$V = 883.4$ (3) Å^3	0.16 × 0.12 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3537 independent reflections
Radiation source: fine-focus sealed tube	1907 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 294$(2) K	$\theta_{max} = 26.4$º
φ and ω scans	$\theta_{min} = 0.9$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -6 \rightarrow 4$
$T_{min} = 0.982$, $T_{max} = 0.991$	$k = -9 \rightarrow 9$
5097 measured reflections	$l = -29 \rightarrow 28$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.2554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.135$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.99$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
3537 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
248 parameters	Extinction correction: ?
Primary atom site location: structure-invariant direct methods	Extinction coefficient: ?
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	1.3675 (5)	1.0676 (3)	0.43874 (9)	0.0579 (6)
H1	1.4940	1.0828	0.4649	0.087*
O2	1.1815 (4)	0.8428 (3)	0.48774 (8)	0.0556 (6)
O3	0.2594 (5)	0.5435 (3)	0.36351 (8)	0.0546 (6)
O4	0.2585 (4)	0.7219 (2)	0.27527 (8)	0.0449 (5)
O5	−0.1359 (4)	1.0118 (2)	0.17115 (7)	0.0389 (5)
O6	−0.3414 (5)	1.2372 (2)	0.22523 (7)	0.0497 (6)
O7	−0.8275 (4)	1.5175 (3)	0.07122 (8)	0.0505 (6)
H7	−0.9377	1.5530	0.0500	0.076*
O8	−0.8096 (4)	1.3542 (3)	−0.00843 (8)	0.0476 (5)
C1	1.1723 (7)	0.9327 (4)	0.44555 (12)	0.0441 (7)
C2	0.9296 (6)	0.8816 (4)	0.40125 (11)	0.0419 (7)
C3	0.9210 (6)	0.9787 (4)	0.35512 (11)	0.0432 (7)
H3	1.0660	1.0776	0.3525	0.052*
C4	0.6972 (6)	0.9293 (4)	0.31276 (11)	0.0426 (7)
H4	0.6922	0.9957	0.2819	0.051*
C5	0.4823 (6)	0.7825 (4)	0.31606 (11)	0.0379 (7)
C6	0.4849 (6)	0.6843 (4)	0.36349 (11)	0.0410 (7)
C7	0.7098 (7)	0.7356 (4)	0.40541 (11)	0.0438 (7)
H7A	0.7141	0.6713	0.4368	0.053*
C8	0.2343 (9)	0.4518 (4)	0.41381 (14)	0.0752 (11)
H8A	0.3895	0.3982	0.4189	0.113*
H8B	0.0578	0.3642	0.4100	0.113*
H8C	0.2386	0.5317	0.4463	0.113*
C9	0.2334 (6)	0.8329 (4)	0.23078 (11)	0.0405 (7)
H9A	0.4022	0.8533	0.2103	0.049*
H9B	0.2139	0.9440	0.2469	0.049*
C10	−0.0230 (6)	0.7462 (3)	0.19106 (11)	0.0401 (7)
H10A	−0.1880	0.7192	0.2126	0.048*
H10B	0.0028	0.6378	0.1740	0.048*
C11	−0.0767 (6)	0.8586 (3)	0.14434 (11)	0.0408 (7)
H11A	0.0891	0.8903	0.1232	0.049*
H11B	−0.2370	0.7962	0.1180	0.049*

C12	−0.2660 (5)	1.1086 (3)	0.13794 (11)	0.0332 (6)
C13	−0.2889 (6)	1.0933 (3)	0.07897 (11)	0.0378 (7)
H13	−0.2030	1.0179	0.0595	0.045*
C14	−0.4406 (6)	1.1908 (3)	0.04883 (11)	0.0386 (7)
H14	−0.4568	1.1795	0.0092	0.046*
C15	−0.5676 (5)	1.3045 (3)	0.07725 (11)	0.0350 (6)
C16	−0.5356 (6)	1.3250 (3)	0.13690 (11)	0.0363 (7)
H16	−0.6171	1.4033	0.1562	0.044*
C17	−0.3841 (6)	1.2299 (3)	0.16731 (10)	0.0347 (6)
C18	−0.7436 (6)	1.3963 (3)	0.04375 (12)	0.0374 (7)
C19	−0.4814 (8)	1.3418 (4)	0.25887 (12)	0.0677 (11)
H19A	−0.4077	1.4619	0.2530	0.102*
H19B	−0.4482	1.3251	0.2985	0.102*
H19C	−0.6820	1.3082	0.2476	0.102*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0599 (15)	0.0580 (14)	0.0537 (14)	0.0173 (12)	−0.0168 (11)	0.0046 (11)
O2	0.0698 (15)	0.0620 (14)	0.0390 (12)	0.0269 (12)	−0.0092 (11)	0.0106 (11)
O3	0.0666 (15)	0.0452 (12)	0.0504 (13)	0.0107 (11)	−0.0021 (11)	0.0133 (10)
O4	0.0524 (13)	0.0423 (11)	0.0416 (12)	0.0164 (10)	−0.0068 (10)	0.0100 (9)
O5	0.0462 (12)	0.0398 (11)	0.0343 (10)	0.0204 (9)	−0.0055 (9)	0.0026 (8)
O6	0.0782 (15)	0.0501 (12)	0.0278 (10)	0.0310 (11)	−0.0003 (10)	0.0019 (9)
O7	0.0586 (15)	0.0558 (13)	0.0449 (12)	0.0321 (11)	−0.0064 (10)	0.0055 (10)
O8	0.0520 (13)	0.0617 (13)	0.0338 (11)	0.0253 (11)	−0.0035 (10)	0.0056 (10)
C1	0.053 (2)	0.0454 (19)	0.0386 (17)	0.0252 (17)	−0.0026 (15)	−0.0010 (14)
C2	0.0489 (19)	0.0464 (18)	0.0355 (16)	0.0242 (15)	−0.0009 (14)	0.0001 (13)
C3	0.0466 (19)	0.0475 (18)	0.0380 (17)	0.0170 (15)	0.0011 (14)	0.0053 (14)
C4	0.052 (2)	0.0465 (18)	0.0333 (16)	0.0204 (16)	0.0000 (14)	0.0073 (13)
C5	0.0439 (18)	0.0413 (16)	0.0327 (15)	0.0211 (14)	−0.0020 (13)	0.0010 (13)
C6	0.051 (2)	0.0372 (16)	0.0376 (17)	0.0177 (15)	0.0023 (14)	0.0026 (13)
C7	0.061 (2)	0.0452 (18)	0.0338 (16)	0.0292 (17)	0.0024 (15)	0.0082 (13)
C8	0.111 (3)	0.053 (2)	0.060 (2)	0.014 (2)	0.004 (2)	0.0254 (18)
C9	0.0412 (18)	0.0460 (17)	0.0377 (16)	0.0174 (14)	−0.0025 (13)	0.0093 (13)
C10	0.0467 (18)	0.0359 (16)	0.0402 (16)	0.0178 (14)	−0.0028 (14)	0.0011 (13)
C11	0.0451 (18)	0.0413 (16)	0.0376 (16)	0.0180 (14)	−0.0047 (13)	−0.0027 (13)
C12	0.0297 (15)	0.0353 (15)	0.0342 (15)	0.0081 (12)	−0.0026 (12)	0.0070 (12)
C13	0.0388 (17)	0.0441 (16)	0.0334 (16)	0.0161 (14)	0.0028 (13)	0.0033 (13)
C14	0.0377 (17)	0.0506 (17)	0.0271 (14)	0.0119 (14)	−0.0029 (13)	0.0044 (13)
C15	0.0316 (16)	0.0383 (15)	0.0358 (16)	0.0103 (13)	0.0001 (12)	0.0054 (12)
C16	0.0411 (17)	0.0331 (15)	0.0353 (16)	0.0112 (13)	0.0009 (13)	0.0031 (12)
C17	0.0408 (17)	0.0341 (15)	0.0293 (15)	0.0094 (13)	0.0016 (13)	0.0046 (12)
C18	0.0364 (17)	0.0370 (16)	0.0402 (17)	0.0120 (13)	−0.0002 (14)	0.0066 (13)
C19	0.120 (3)	0.056 (2)	0.0395 (18)	0.042 (2)	0.020 (2)	0.0039 (16)

Geometric parameters (Å, °)

O1—C1	1.277 (3)	C8—H8A	0.9600

O1—H1	0.8200	C8—H8B	0.9600
O2—C1	1.269 (3)	C8—H8C	0.9600
O3—C6	1.365 (3)	C9—C10	1.495 (4)
O3—C8	1.432 (3)	C9—H9A	0.9700
O4—C5	1.369 (3)	C9—H9B	0.9700
O4—C9	1.434 (3)	C10—C11	1.513 (3)
O5—C12	1.363 (3)	C10—H10A	0.9700
O5—C11	1.441 (3)	C10—H10B	0.9700
O6—C17	1.363 (3)	C11—H11A	0.9700
O6—C19	1.437 (3)	C11—H11B	0.9700
O7—C18	1.294 (3)	C12—C13	1.383 (3)
O7—H7	0.8200	C12—C17	1.411 (3)
O8—C18	1.252 (3)	C13—C14	1.390 (3)
C1—C2	1.477 (4)	C13—H13	0.9300
C2—C3	1.380 (4)	C14—C15	1.383 (4)
C2—C7	1.390 (4)	C14—H14	0.9300
C3—C4	1.386 (4)	C15—C16	1.399 (3)
C3—H3	0.9300	C15—C18	1.476 (3)
C4—C5	1.377 (4)	C16—C17	1.379 (3)
C4—H4	0.9300	C16—H16	0.9300
C5—C6	1.408 (4)	C19—H19A	0.9600
C6—C7	1.384 (4)	C19—H19B	0.9600
C7—H7A	0.9300	C19—H19C	0.9600
C1—O1—H1	109.5	C9—C10—C11	112.2 (2)
C6—O3—C8	117.7 (2)	C9—C10—H10A	109.2
C5—O4—C9	116.2 (2)	C11—C10—H10A	109.2
C12—O5—C11	117.88 (19)	C9—C10—H10B	109.2
C17—O6—C19	118.7 (2)	C11—C10—H10B	109.2
C18—O7—H7	109.5	H10A—C10—H10B	107.9
O2—C1—O1	122.9 (3)	O5—C11—C10	107.4 (2)
O2—C1—C2	120.0 (3)	O5—C11—H11A	110.2
O1—C1—C2	117.1 (3)	C10—C11—H11A	110.2
C3—C2—C7	119.6 (3)	O5—C11—H11B	110.2
C3—C2—C1	119.9 (3)	C10—C11—H11B	110.2
C7—C2—C1	120.5 (3)	H11A—C11—H11B	108.5
C2—C3—C4	120.2 (3)	O5—C12—C13	124.3 (2)
C2—C3—H3	119.9	O5—C12—C17	115.9 (2)
C4—C3—H3	119.9	C13—C12—C17	119.8 (2)
C5—C4—C3	120.4 (3)	C12—C13—C14	120.0 (3)
C5—C4—H4	119.8	C12—C13—H13	120.0
C3—C4—H4	119.8	C14—C13—H13	120.0
O4—C5—C4	124.1 (2)	C15—C14—C13	120.6 (2)
O4—C5—C6	115.9 (3)	C15—C14—H14	119.7
C4—C5—C6	120.0 (3)	C13—C14—H14	119.7
O3—C6—C7	125.5 (3)	C14—C15—C16	119.5 (2)
O3—C6—C5	115.6 (3)	C14—C15—C18	118.9 (2)
C7—C6—C5	118.8 (3)	C16—C15—C18	121.6 (2)
C6—C7—C2	120.9 (3)	C17—C16—C15	120.4 (2)
C6—C7—H7A	119.5	C17—C16—H16	119.8

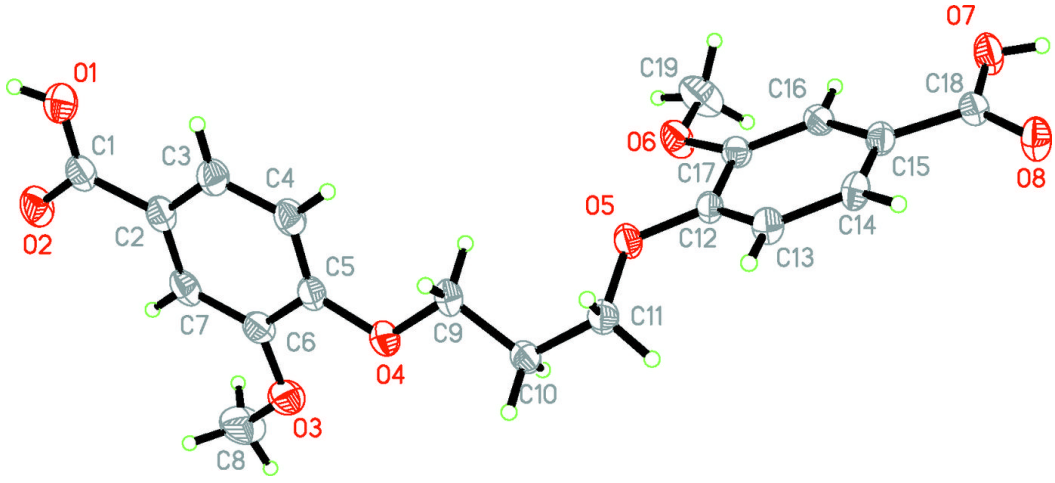
C2—C7—H7A	119.5	C15—C16—H16	119.8
O3—C8—H8A	109.5	O6—C17—C16	125.7 (2)
O3—C8—H8B	109.5	O6—C17—C12	114.7 (2)
H8A—C8—H8B	109.5	C16—C17—C12	119.6 (2)
O3—C8—H8C	109.5	O8—C18—O7	122.9 (2)
H8A—C8—H8C	109.5	O8—C18—C15	120.4 (2)
H8B—C8—H8C	109.5	O7—C18—C15	116.7 (2)
O4—C9—C10	108.4 (2)	O6—C19—H19A	109.5
O4—C9—H9A	110.0	O6—C19—H19B	109.5
C10—C9—H9A	110.0	H19A—C19—H19B	109.5
O4—C9—H9B	110.0	O6—C19—H19C	109.5
C10—C9—H9B	110.0	H19A—C19—H19C	109.5
H9A—C9—H9B	108.4	H19B—C19—H19C	109.5
O2—C1—C2—C3	−179.4 (2)	C12—O5—C11—C10	−162.3 (2)
O1—C1—C2—C3	−0.1 (4)	C9—C10—C11—O5	−63.3 (3)
O2—C1—C2—C7	0.1 (4)	C11—O5—C12—C13	−14.9 (4)
O1—C1—C2—C7	179.5 (3)	C11—O5—C12—C17	164.6 (2)
C7—C2—C3—C4	−1.0 (4)	O5—C12—C13—C14	176.1 (2)
C1—C2—C3—C4	178.5 (2)	C17—C12—C13—C14	−3.3 (4)
C2—C3—C4—C5	−0.4 (4)	C12—C13—C14—C15	0.5 (4)
C9—O4—C5—C4	−8.3 (4)	C13—C14—C15—C16	1.9 (4)
C9—O4—C5—C6	171.9 (2)	C13—C14—C15—C18	−175.7 (2)
C3—C4—C5—O4	−177.9 (2)	C14—C15—C16—C17	−1.5 (4)
C3—C4—C5—C6	1.8 (4)	C18—C15—C16—C17	176.1 (2)
C8—O3—C6—C7	7.6 (4)	C19—O6—C17—C16	5.1 (4)
C8—O3—C6—C5	−172.6 (2)	C19—O6—C17—C12	−172.9 (3)
O4—C5—C6—O3	−1.7 (3)	C15—C16—C17—O6	−179.2 (2)
C4—C5—C6—O3	178.5 (2)	C15—C16—C17—C12	−1.3 (4)
O4—C5—C6—C7	178.0 (2)	O5—C12—C17—O6	2.4 (3)
C4—C5—C6—C7	−1.7 (4)	C13—C12—C17—O6	−178.2 (2)
O3—C6—C7—C2	−180.0 (3)	O5—C12—C17—C16	−175.8 (2)
C5—C6—C7—C2	0.3 (4)	C13—C12—C17—C16	3.7 (4)
C3—C2—C7—C6	1.1 (4)	C14—C15—C18—O8	9.9 (4)
C1—C2—C7—C6	−178.5 (2)	C16—C15—C18—O8	−167.6 (2)
C5—O4—C9—C10	−179.4 (2)	C14—C15—C18—O7	−171.3 (2)
O4—C9—C10—C11	176.8 (2)	C16—C15—C18—O7	11.2 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.81	2.618 (3)	166
O7—H7···O8^{ii}	0.82	1.84	2.658 (3)	174

Symmetry codes: (i) −*x*+3, −*y*+2, −*z*+1; (ii) −*x*−2, −*y*+3, −*z*.

Fig. 1


O1
C1
C3
C4
O2
C2
C5
C7
C6
O4
C9
C10
O3
C8
C11
O5
C12
C13
C14
C15
C16
C17
O6
C19
C18
O7
O8