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3-(1,3-Benzodioxol-5-yl)-3H-benzo[*f*]isobenzofuran-1-oneS. Thenmozhi,^a A. SubbiahPandi,^{a*} J. Arulclement^b and A. K. MohanaKrishnan^b^aDepartment of Physics, Presidency College (Autonomous), Chennai 600 005, India, and ^bDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

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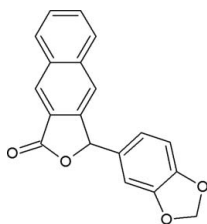
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.046; wR factor = 0.137; data-to-parameter ratio = 20.4.

In the title compound, $\text{C}_{19}\text{H}_{12}\text{O}_4$, the dioxole ring adopts a flattened envelope conformation with the methylene C at the flap [deviation = $0.104(2)$ Å]. The benzene ring of the benzodioxole ring system makes a dihedral angle of $76.45(5)^\circ$ with the planar [maximum deviation = $0.016(1)$ Å] 3H-benzo[*f*]isobenzofuran-1-one ring system. In the crystal structure, the molecules are linked into $C(5)$ chains running along the b axis by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds. In addition, $\text{C}-\text{H}\cdots\pi$ interactions are observed.

Related literature

For the biological activity of benzofuran compounds, see: Howlett *et al.* (1999); Twyman & Allsop (1999); Valerga *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{19}\text{H}_{12}\text{O}_4$ $M_r = 304.29$

Monoclinic, $P2_1/c$
 $a = 7.8617(3)$ Å
 $b = 12.0417(4)$ Å
 $c = 15.0214(5)$ Å
 $\beta = 95.848(2)^\circ$
 $V = 1414.65(9)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.21 \times 0.19 \times 0.17$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.979$, $T_{\max} = 0.983$

19039 measured reflections
 4251 independent reflections
 3370 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.137$
 $S = 1.02$
 4251 reflections

208 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C13–C18 ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C12}-\text{H12}\cdots\text{O1}^{\text{i}}$	0.98	2.39	3.3277 (16)	159
$\text{C3}-\text{H3}\cdots\text{Cg1}^{\text{ii}}$	0.93	2.84	3.6021 (15)	140

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 2, -y + 2, -z$.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

ST and ASP thank Dr Babu Varghese, SAIF, IIT-Madras, Chennai, India, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5047).

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supporting information

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3-(1,3-Benzodioxol-5-yl)-3H-benzo[f]isobenzofuran-1-one

S. Thenmozhi, A. SubbiahPandi, J. Arulclement and A. K. MohanaKrishnan

S1. Comment

Molecules containing a benzofuran ring system have attracted considerable interest in view of their biological and pharmacological properties (Howlett *et al.*, 1999; Twyman & Allsop, 1999). Furan compounds exhibit antibacterial and antifungal activities (Valerga *et al.*, 2009). Against this background and in order to obtain detailed information on molecular conformation in the solid state, an X-ray crystallographic study of the title compound has been carried out and the results are presented here.

The 3H-benzo[f]isobenzofuran-1-one ring system is essentially planar, with a maximum deviation of 0.016 (1) Å for atom C12. In the 1,3-benzodioxole ring system, the dioxole ring adopts a flattened envelope conformation with the methylene C at the flap [deviation = 0.104 (2) Å]. The benzene ring of the benzodioxole ring system makes a dihedral angle of 76.45 (5)° with the 3H-benzo[f]isobenzofuran-1-one ring system.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 1) involving atoms C12 and O1, link the molecules into chains which run parallel to the *b* axis and can be described by a graph set motif of C(5). In addition, the crystal packing is stabilized by C—H··· π interactions involving the C13–C18 ring.

S2. Experimental

NaBH₄ (1.6 g, 43.75 mmol) was carefully added in small portions to a solution of keto acid (3.5 g, 10.93 mmol) in THF-EtOH (2:5) at 273 K. The reaction mixture was refluxed for 12 h and then poured into ice water (200 ml). The reaction mixture was acidified using HCl (pH = 2-3) and then stirred for 0.5 h at room temperature. The solid formed was filtered and washed with methanol to afford lactone as a colourless solid.

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C–H distances fixed in the range 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

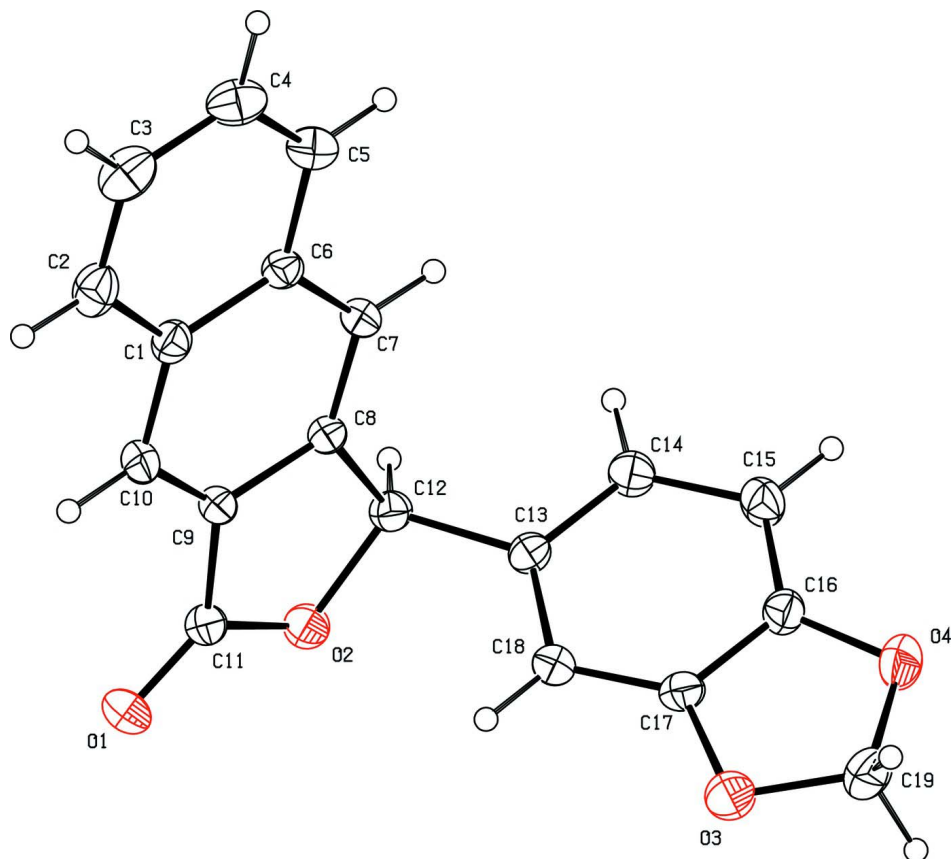


Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

3-(1,3-Benzodioxol-5-yl)-3H-benzo[f]isobenzofuran-1-one

Crystal data

$C_{19}H_{12}O_4$
 $M_r = 304.29$
 Monoclinic, $P2_1/c$
 Hall symbol: $-P\ 2ybc$
 $a = 7.8617\ (3)\ \text{\AA}$
 $b = 12.0417\ (4)\ \text{\AA}$
 $c = 15.0214\ (5)\ \text{\AA}$
 $\beta = 95.848\ (2)^\circ$
 $V = 1414.65\ (9)\ \text{\AA}^3$
 $Z = 4$

$F(000) = 632$
 $D_x = 1.429\ \text{Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 4251 reflections
 $\theta = 2.2\text{--}30.4^\circ$
 $\mu = 0.10\ \text{mm}^{-1}$
 $T = 293\ \text{K}$
 Block, colourless
 $0.21 \times 0.19 \times 0.17\ \text{mm}$

Data collection

Bruker Kappa APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans

Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.979$, $T_{\max} = 0.983$
 19039 measured reflections
 4251 independent reflections
 3370 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 30.4^\circ$, $\theta_{\text{min}} = 2.2^\circ$
 $h = -11 \rightarrow 10$

$k = -17 \rightarrow 16$
 $l = -21 \rightarrow 13$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.137$
 $S = 1.02$
 4251 reflections
 208 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.2801P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.50740 (15)	1.28238 (9)	0.19129 (8)	0.0587 (3)
O2	0.58783 (11)	1.12957 (8)	0.26946 (6)	0.0419 (2)
O3	1.20824 (14)	1.15928 (9)	0.41662 (8)	0.0588 (3)
O4	1.29651 (13)	0.98350 (9)	0.46223 (8)	0.0548 (3)
C1	0.70723 (14)	1.07497 (10)	-0.02366 (7)	0.0333 (2)
C2	0.71799 (18)	1.09700 (12)	-0.11570 (9)	0.0443 (3)
H2	0.6798	1.1648	-0.1397	0.053*
C3	0.7832 (2)	1.02046 (14)	-0.16932 (9)	0.0503 (4)
H3	0.7902	1.0364	-0.2294	0.060*
C4	0.84016 (19)	0.91727 (13)	-0.13420 (9)	0.0479 (3)
H4	0.8842	0.8652	-0.1714	0.057*
C5	0.83164 (17)	0.89258 (11)	-0.04622 (9)	0.0401 (3)
H5	0.8700	0.8239	-0.0241	0.048*
C6	0.76513 (14)	0.97022 (9)	0.01198 (7)	0.0307 (2)
C7	0.75593 (14)	0.94554 (9)	0.10366 (7)	0.0313 (2)
H7	0.7938	0.8775	0.1274	0.038*
C8	0.69034 (13)	1.02365 (9)	0.15634 (7)	0.0289 (2)
C9	0.63280 (14)	1.12596 (9)	0.12091 (8)	0.0324 (2)
C10	0.64042 (16)	1.15336 (10)	0.03305 (8)	0.0369 (3)
H10	0.6023	1.2222	0.0112	0.044*
C11	0.56804 (15)	1.19097 (11)	0.19281 (9)	0.0396 (3)
C12	0.66602 (15)	1.02200 (10)	0.25451 (7)	0.0329 (2)

H12	0.5871	0.9622	0.2667	0.039*
C13	0.83094 (15)	1.00982 (10)	0.31446 (7)	0.0310 (2)
C14	0.88263 (17)	0.90475 (10)	0.34263 (9)	0.0389 (3)
H14	0.8110	0.8446	0.3280	0.047*
C15	1.04087 (17)	0.88686 (11)	0.39288 (9)	0.0420 (3)
H15	1.0765	0.8161	0.4114	0.050*
C16	1.13967 (16)	0.97803 (11)	0.41324 (8)	0.0364 (3)
C17	1.08753 (16)	1.08282 (10)	0.38590 (8)	0.0355 (2)
C18	0.93465 (15)	1.10207 (10)	0.33622 (8)	0.0351 (2)
H18	0.9013	1.1732	0.3178	0.042*
C19	1.34790 (18)	1.09710 (13)	0.45929 (10)	0.0497 (3)
H19A	1.3783	1.1248	0.5195	0.060*
H19B	1.4468	1.1041	0.4260	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0624 (7)	0.0454 (6)	0.0653 (7)	0.0244 (5)	-0.0085 (5)	-0.0180 (5)
O2	0.0383 (5)	0.0475 (5)	0.0398 (4)	0.0108 (4)	0.0030 (4)	-0.0106 (4)
O3	0.0477 (6)	0.0393 (5)	0.0841 (8)	-0.0062 (4)	-0.0198 (5)	-0.0088 (5)
O4	0.0438 (5)	0.0521 (6)	0.0637 (7)	0.0030 (4)	-0.0172 (5)	0.0006 (5)
C1	0.0314 (5)	0.0338 (6)	0.0336 (5)	-0.0029 (4)	-0.0020 (4)	0.0019 (4)
C2	0.0467 (7)	0.0483 (7)	0.0367 (6)	-0.0045 (6)	-0.0013 (5)	0.0095 (5)
C3	0.0534 (8)	0.0644 (9)	0.0342 (6)	-0.0127 (7)	0.0097 (5)	0.0010 (6)
C4	0.0511 (8)	0.0527 (8)	0.0424 (7)	-0.0085 (6)	0.0165 (6)	-0.0114 (6)
C5	0.0432 (6)	0.0367 (6)	0.0415 (6)	-0.0020 (5)	0.0094 (5)	-0.0059 (5)
C6	0.0292 (5)	0.0291 (5)	0.0335 (5)	-0.0028 (4)	0.0014 (4)	-0.0027 (4)
C7	0.0343 (5)	0.0248 (5)	0.0342 (5)	0.0013 (4)	0.0011 (4)	-0.0002 (4)
C8	0.0271 (5)	0.0266 (5)	0.0322 (5)	-0.0010 (4)	-0.0010 (4)	-0.0023 (4)
C9	0.0306 (5)	0.0267 (5)	0.0385 (5)	0.0038 (4)	-0.0037 (4)	-0.0043 (4)
C10	0.0391 (6)	0.0283 (5)	0.0412 (6)	0.0044 (4)	-0.0056 (5)	0.0026 (4)
C11	0.0333 (6)	0.0383 (6)	0.0453 (6)	0.0072 (5)	-0.0056 (5)	-0.0111 (5)
C12	0.0319 (5)	0.0336 (6)	0.0331 (5)	0.0005 (4)	0.0026 (4)	-0.0039 (4)
C13	0.0328 (5)	0.0335 (5)	0.0270 (5)	-0.0011 (4)	0.0037 (4)	-0.0009 (4)
C14	0.0402 (6)	0.0319 (6)	0.0440 (6)	-0.0052 (5)	0.0016 (5)	0.0033 (5)
C15	0.0453 (7)	0.0334 (6)	0.0463 (7)	0.0007 (5)	-0.0004 (5)	0.0083 (5)
C16	0.0364 (6)	0.0401 (6)	0.0320 (5)	0.0031 (5)	0.0000 (4)	0.0004 (4)
C17	0.0372 (6)	0.0325 (6)	0.0363 (5)	-0.0029 (4)	0.0010 (4)	-0.0061 (4)
C18	0.0385 (6)	0.0290 (5)	0.0373 (5)	0.0011 (4)	0.0005 (4)	-0.0013 (4)
C19	0.0391 (7)	0.0562 (9)	0.0515 (8)	-0.0040 (6)	-0.0063 (6)	-0.0052 (6)

Geometric parameters (Å, °)

O1—C11	1.1988 (15)	C7—H7	0.93
O2—C11	1.3639 (17)	C8—C9	1.3988 (15)
O2—C12	1.4610 (14)	C8—C12	1.5063 (15)
O3—C17	1.3682 (15)	C9—C10	1.3675 (17)
O3—C19	1.4260 (18)	C9—C11	1.4665 (16)

O4—C16	1.3716 (15)	C10—H10	0.93
O4—C19	1.4283 (18)	C12—C13	1.5085 (16)
C1—C10	1.4085 (17)	C12—H12	0.98
C1—C2	1.4188 (17)	C13—C14	1.3820 (17)
C1—C6	1.4266 (16)	C13—C18	1.3969 (16)
C2—C3	1.358 (2)	C14—C15	1.4044 (18)
C2—H2	0.93	C14—H14	0.93
C3—C4	1.405 (2)	C15—C16	1.3615 (18)
C3—H3	0.93	C15—H15	0.93
C4—C5	1.3628 (19)	C16—C17	1.3765 (17)
C4—H4	0.93	C17—C18	1.3684 (17)
C5—C6	1.4160 (16)	C18—H18	0.93
C5—H5	0.93	C19—H19A	0.97
C6—C7	1.4178 (15)	C19—H19B	0.97
C7—C8	1.3638 (15)		
C11—O2—C12	111.37 (9)	O1—C11—O2	121.65 (12)
C17—O3—C19	105.91 (11)	O1—C11—C9	130.04 (13)
C16—O4—C19	105.73 (10)	O2—C11—C9	108.31 (10)
C10—C1—C2	121.80 (11)	O2—C12—C8	103.66 (9)
C10—C1—C6	119.34 (10)	O2—C12—C13	110.11 (9)
C2—C1—C6	118.86 (11)	C8—C12—C13	113.49 (9)
C3—C2—C1	120.96 (13)	O2—C12—H12	109.8
C3—C2—H2	119.5	C8—C12—H12	109.8
C1—C2—H2	119.5	C13—C12—H12	109.8
C2—C3—C4	120.17 (12)	C14—C13—C18	120.66 (11)
C2—C3—H3	119.9	C14—C13—C12	118.67 (10)
C4—C3—H3	119.9	C18—C13—C12	120.54 (10)
C5—C4—C3	120.77 (13)	C13—C14—C15	121.45 (12)
C5—C4—H4	119.6	C13—C14—H14	119.3
C3—C4—H4	119.6	C15—C14—H14	119.3
C4—C5—C6	120.83 (13)	C16—C15—C14	116.80 (11)
C4—C5—H5	119.6	C16—C15—H15	121.6
C6—C5—H5	119.6	C14—C15—H15	121.6
C7—C6—C5	121.33 (11)	C15—C16—O4	128.34 (12)
C7—C6—C1	120.26 (10)	C15—C16—C17	121.77 (11)
C5—C6—C1	118.41 (11)	O4—C16—C17	109.88 (11)
C8—C7—C6	118.60 (10)	O3—C17—C18	127.62 (12)
C8—C7—H7	120.7	O3—C17—C16	109.94 (11)
C6—C7—H7	120.7	C18—C17—C16	122.43 (11)
C7—C8—C9	120.89 (10)	C17—C18—C13	116.89 (11)
C7—C8—C12	130.70 (10)	C17—C18—H18	121.6
C9—C8—C12	108.41 (9)	C13—C18—H18	121.6
C10—C9—C8	122.37 (11)	O3—C19—O4	108.03 (11)
C10—C9—C11	129.39 (11)	O3—C19—H19A	110.1
C8—C9—C11	108.24 (10)	O4—C19—H19A	110.1
C9—C10—C1	118.54 (11)	O3—C19—H19B	110.1
C9—C10—H10	120.7	O4—C19—H19B	110.1

C1—C10—H10	120.7	H19A—C19—H19B	108.4
C10—C1—C2—C3	179.83 (13)	C11—O2—C12—C8	-0.87 (12)
C6—C1—C2—C3	-0.45 (19)	C11—O2—C12—C13	120.86 (11)
C1—C2—C3—C4	0.5 (2)	C7—C8—C12—O2	-179.21 (11)
C2—C3—C4—C5	-0.3 (2)	C9—C8—C12—O2	1.19 (12)
C3—C4—C5—C6	0.0 (2)	C7—C8—C12—C13	61.35 (16)
C4—C5—C6—C7	-179.88 (12)	C9—C8—C12—C13	-118.25 (11)
C4—C5—C6—C1	-0.01 (18)	O2—C12—C13—C14	150.18 (11)
C10—C1—C6—C7	-0.20 (16)	C8—C12—C13—C14	-94.13 (13)
C2—C1—C6—C7	-179.92 (11)	O2—C12—C13—C18	-33.93 (14)
C10—C1—C6—C5	179.93 (11)	C8—C12—C13—C18	81.76 (13)
C2—C1—C6—C5	0.21 (17)	C18—C13—C14—C15	-0.74 (19)
C5—C6—C7—C8	-179.90 (11)	C12—C13—C14—C15	175.14 (11)
C1—C6—C7—C8	0.23 (16)	C13—C14—C15—C16	0.7 (2)
C6—C7—C8—C9	0.16 (16)	C14—C15—C16—O4	178.80 (13)
C6—C7—C8—C12	-179.39 (11)	C14—C15—C16—C17	-0.1 (2)
C7—C8—C9—C10	-0.61 (17)	C19—O4—C16—C15	176.89 (14)
C12—C8—C9—C10	179.03 (11)	C19—O4—C16—C17	-4.11 (15)
C7—C8—C9—C11	179.26 (10)	C19—O3—C17—C18	-176.17 (13)
C12—C8—C9—C11	-1.10 (13)	C19—O3—C17—C16	4.60 (15)
C8—C9—C10—C1	0.63 (18)	C15—C16—C17—O3	178.77 (12)
C11—C9—C10—C1	-179.21 (11)	O4—C16—C17—O3	-0.31 (15)
C2—C1—C10—C9	179.49 (12)	C15—C16—C17—C18	-0.5 (2)
C6—C1—C10—C9	-0.22 (17)	O4—C16—C17—C18	-179.59 (12)
C12—O2—C11—O1	-179.35 (12)	O3—C17—C18—C13	-178.66 (12)
C12—O2—C11—C9	0.24 (13)	C16—C17—C18—C13	0.49 (18)
C10—C9—C11—O1	0.0 (2)	C14—C13—C18—C17	0.13 (17)
C8—C9—C11—O1	-179.90 (14)	C12—C13—C18—C17	-175.68 (10)
C10—C9—C11—O2	-179.59 (12)	C17—O3—C19—O4	-7.07 (15)
C8—C9—C11—O2	0.56 (13)	C16—O4—C19—O3	6.88 (15)

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

Cg1 is the centroid of the C13–C18 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12—H12 \cdots O1 ⁱ	0.98	2.39	3.3277 (16)	159
C3—H3 \cdots Cg1 ⁱⁱ	0.93	2.84	3.6021 (15)	140

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