

3-(2,4-Dichlorophenyl)-2-oxo-1-oxaspiro[4.5]dec-3-en-4-yl 2-methyl-prop-2-enoate

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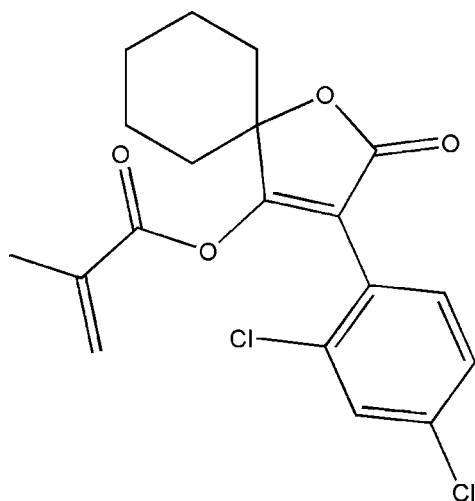
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 19.3.

In the title molecule, $\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{O}_4$, the cyclohexane ring adopts a chair conformation. The furan ring is essentially planar and forms a dihedral angle of $82.1(1)^\circ$ with the benzene ring. In the crystal, weak $\text{C}-\text{H}\cdots\text{O}$ interactions are present.

Related literature

For the potential biological activity of the title compound and the crystal structures of related compounds, see: Bretschneider *et al.* (2003). For the synthesis, see: Lu *et al.* (2008).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{O}_4$
 $M_r = 381.23$
 Monoclinic, $P2_1/n$
 $a = 10.759(1)$ Å
 $b = 11.8778(11)$ Å
 $c = 15.0130(15)$ Å
 $\beta = 107.047(4)^\circ$

$V = 1834.3(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 113$ K
 $0.22 \times 0.20 \times 0.14$ mm

Data collection

Rigaku Saturn CCD diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.922$, $T_{\max} = 0.950$

17672 measured reflections
 4371 independent reflections
 3362 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.092$
 $S = 1.03$
 4371 reflections

227 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.55$ e Å⁻³
 $\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C11}-\text{H11}\cdots\text{O1}^{\text{i}}$	0.95	2.52	3.2470 (17)	133
$\text{C18}-\text{H18B}\cdots\text{O1}^{\text{ii}}$	0.95	2.56	3.4486 (17)	156

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5451).

References

- Bretschneider, T., Benet-Buchholz, J., Fischer, R. & Nauen, R. (2003). *Chimia*, **57**, 697–701.
 Lu, Y., Tao, J. Z. & Zhang, Z. R. (2008). *Chem. Intermed.* **10**, 25–28.
 Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2012). E68, o1531 [https://doi.org/10.1107/S1600536812016340]

3-(2,4-Dichlorophenyl)-2-oxo-1-oxaspiro[4.5]dec-3-en-4-yl 2-methylprop-2-enoate

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S1. Comment

The title compound (I) was synthesized as a new compound with potential biological activity (Bretschneider *et al.*, 2003). We report herein its crystal structure.

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Bretschneider *et al.*, 2003). The cyclohexane ring (C4—C9) adopts a chair conformation. The furan ring (O2/C1-C4) plane forms a dihedral angle of 82.1 (1)° with the benzene ring (C10—C15). In the crystal, weak intermolecular C—H···O hydrogen bonds are present.

S2. Experimental

The synthesis followed the procedure of Lu *et al.* (2008). In a flask equipped with stirrer and reflux condenser, 3-(2,4-dichlorophenyl)-2-oxo-1-oxaspiro[4.5]dec-3-ene-4-ol 3.13 g (10.0 mmol), and triethylamine 5 ml was mixed in dichloromethane (30 ml), at 273–278 K. The mixture was stirred, then methacryloyl chloride 1.25 g (12.0 mmol) was added dropwise for 1 h, then the mixture was left at room temperature for 3 h. The mixture was then washed with 1% HCl (60 ml) and water (60 ml), and the organic layer was dried over sodium sulfate. Excess dichloromethane was removed on a water vacuum pump to obtain an oily colorless product. The product was crystallized from methanol to afford the title compound 3.39 g (89% yield). Single crystals suitable for X-ray measurements were obtained from a solution of the title compound in acetone and methanol at room temperature.

S3. Refinement

H atoms were placed in calculated positions, with C—H = 0.95–0.99 Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$ for methyl C atoms.

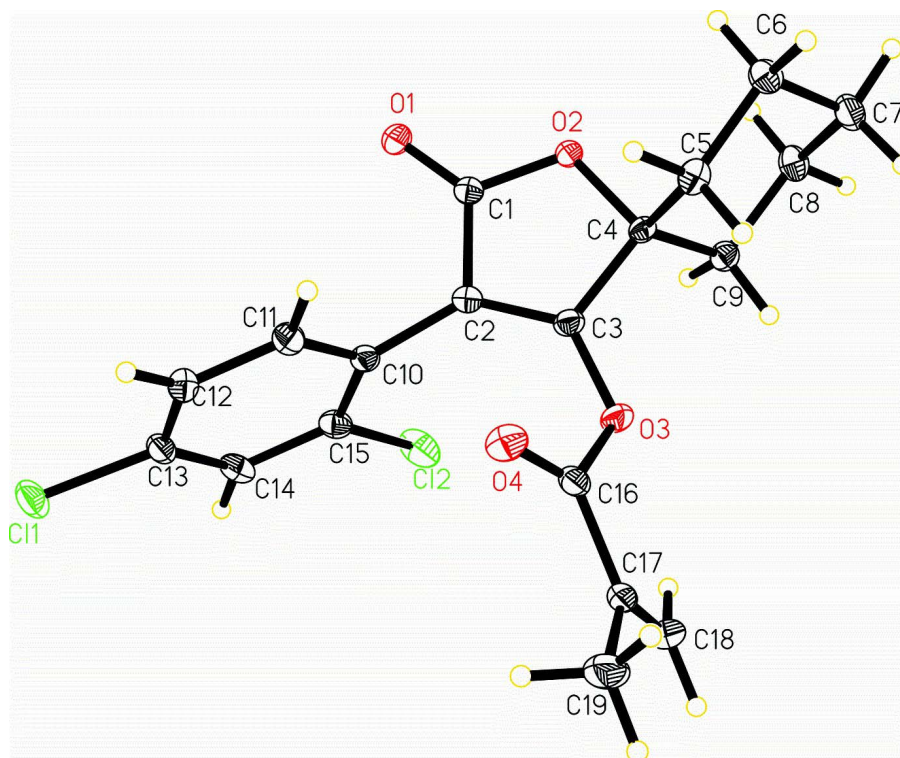


Figure 1

View of the title compound with displacement ellipsoids drawn at the 40% probability level.

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Crystal data

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 Monoclinic, $P2_1/n$
 Hall symbol: -P 2yn
 $a = 10.759$ (1) Å
 $b = 11.8778$ (11) Å
 $c = 15.0130$ (15) Å
 $\beta = 107.047$ (4)°
 $V = 1834.3$ (3) Å³
 $Z = 4$

$F(000) = 792$
 $D_x = 1.381$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 6104 reflections
 $\theta = 1.4$ – 28.1 °
 $\mu = 0.37$ mm⁻¹
 $T = 113$ K
 Prism, colorless
 $0.22 \times 0.20 \times 0.14$ mm

Data collection

Rigaku Saturn CCD
 diffractometer
 Radiation source: rotating anode
 Multilayer monochromator
 Detector resolution: 14.63 pixels mm⁻¹
 ω and ϕ scans
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.922$, $T_{\max} = 0.950$

17672 measured reflections
 4371 independent reflections
 3362 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 27.9$ °, $\theta_{\min} = 2.1$ °
 $h = -13 \rightarrow 14$
 $k = -14 \rightarrow 15$
 $l = -19 \rightarrow 19$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.092$ $S = 1.03$

4371 reflections

227 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0557P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.002$ $\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$ *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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.51046 (3)	0.62323 (3)	1.13695 (2)	0.02998 (11)
Cl2	0.37184 (4)	0.19570 (3)	1.04198 (3)	0.03410 (12)
O1	0.00843 (9)	0.29580 (7)	1.08091 (6)	0.0212 (2)
O2	-0.07870 (8)	0.18353 (7)	0.95872 (6)	0.01666 (19)
O3	0.10997 (9)	0.21926 (8)	0.80025 (7)	0.0243 (2)
O4	0.10974 (10)	0.40685 (8)	0.77325 (7)	0.0279 (2)
C1	0.00933 (12)	0.26065 (10)	1.00566 (9)	0.0162 (3)
C2	0.09833 (12)	0.28979 (10)	0.95101 (9)	0.0174 (3)
C3	0.05816 (13)	0.23083 (10)	0.87256 (9)	0.0182 (3)
C4	-0.06139 (12)	0.16271 (10)	0.86751 (8)	0.0159 (3)
C5	-0.18169 (13)	0.20806 (10)	0.79466 (9)	0.0202 (3)
H5A	-0.1945	0.2881	0.8082	0.024*
H5B	-0.1682	0.2040	0.7323	0.024*
C6	-0.30301 (13)	0.14107 (11)	0.79411 (10)	0.0234 (3)
H6A	-0.3215	0.1509	0.8545	0.028*
H6B	-0.3785	0.1700	0.7443	0.028*
C7	-0.28405 (14)	0.01632 (11)	0.77779 (10)	0.0252 (3)
H7A	-0.3627	-0.0260	0.7794	0.030*
H7B	-0.2720	0.0060	0.7154	0.030*
C8	-0.16622 (14)	-0.02997 (11)	0.85182 (10)	0.0239 (3)
H8A	-0.1534	-0.1099	0.8379	0.029*
H8B	-0.1826	-0.0268	0.9133	0.029*
C9	-0.04271 (13)	0.03628 (10)	0.85624 (9)	0.0194 (3)
H9A	-0.0183	0.0230	0.7984	0.023*
H9B	0.0293	0.0089	0.9094	0.023*

C10	0.20514 (12)	0.37111 (10)	0.98884 (9)	0.0176 (3)
C11	0.17792 (13)	0.48609 (11)	0.98462 (9)	0.0213 (3)
H11	0.0932	0.5114	0.9514	0.026*
C12	0.27197 (13)	0.56429 (11)	1.02792 (9)	0.0223 (3)
H12	0.2523	0.6424	1.0241	0.027*
C13	0.39456 (13)	0.52694 (11)	1.07668 (9)	0.0211 (3)
C14	0.42685 (13)	0.41434 (11)	1.08054 (10)	0.0225 (3)
H14	0.5123	0.3898	1.1127	0.027*
C15	0.33101 (13)	0.33740 (11)	1.03600 (9)	0.0205 (3)
C16	0.14479 (13)	0.31519 (11)	0.75941 (9)	0.0208 (3)
C17	0.22568 (13)	0.28559 (12)	0.69809 (9)	0.0243 (3)
C18	0.28787 (15)	0.18689 (13)	0.70703 (12)	0.0344 (4)
H18A	0.2806	0.1346	0.7531	0.041*
H18B	0.3394	0.1690	0.6673	0.041*
C19	0.23706 (16)	0.37669 (14)	0.63240 (11)	0.0375 (4)
H19A	0.2959	0.3523	0.5972	0.056*
H19B	0.2716	0.4449	0.6677	0.056*
H19C	0.1511	0.3926	0.5892	0.056*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02639 (19)	0.0348 (2)	0.02668 (19)	−0.01427 (15)	0.00461 (15)	−0.00520 (14)
Cl2	0.02374 (19)	0.02107 (19)	0.0554 (3)	0.00275 (13)	0.00842 (18)	0.00714 (15)
O1	0.0225 (5)	0.0261 (5)	0.0155 (5)	−0.0015 (4)	0.0066 (4)	−0.0027 (4)
O2	0.0173 (4)	0.0202 (4)	0.0135 (4)	−0.0023 (3)	0.0061 (4)	−0.0013 (3)
O3	0.0303 (5)	0.0248 (5)	0.0243 (5)	−0.0074 (4)	0.0182 (4)	−0.0054 (4)
O4	0.0303 (6)	0.0293 (5)	0.0268 (5)	0.0020 (4)	0.0127 (5)	0.0018 (4)
C1	0.0153 (6)	0.0157 (6)	0.0174 (6)	0.0022 (5)	0.0043 (5)	0.0021 (5)
C2	0.0163 (6)	0.0183 (6)	0.0184 (6)	0.0002 (5)	0.0064 (5)	0.0008 (5)
C3	0.0198 (6)	0.0181 (6)	0.0193 (6)	−0.0007 (5)	0.0100 (5)	−0.0001 (5)
C4	0.0184 (6)	0.0180 (6)	0.0131 (6)	−0.0013 (5)	0.0073 (5)	−0.0007 (5)
C5	0.0244 (7)	0.0189 (6)	0.0162 (6)	0.0027 (5)	0.0041 (5)	0.0018 (5)
C6	0.0173 (6)	0.0295 (7)	0.0205 (7)	0.0027 (5)	0.0010 (5)	0.0002 (5)
C7	0.0225 (7)	0.0275 (7)	0.0236 (7)	−0.0063 (6)	0.0036 (6)	−0.0028 (6)
C8	0.0260 (7)	0.0177 (6)	0.0268 (7)	−0.0038 (5)	0.0061 (6)	−0.0008 (5)
C9	0.0197 (6)	0.0177 (6)	0.0208 (7)	0.0011 (5)	0.0060 (5)	−0.0013 (5)
C10	0.0169 (6)	0.0216 (6)	0.0153 (6)	−0.0021 (5)	0.0064 (5)	−0.0009 (5)
C11	0.0185 (6)	0.0227 (7)	0.0218 (7)	−0.0004 (5)	0.0047 (5)	−0.0011 (5)
C12	0.0243 (7)	0.0199 (7)	0.0237 (7)	−0.0025 (5)	0.0085 (6)	−0.0032 (5)
C13	0.0204 (6)	0.0261 (7)	0.0172 (6)	−0.0087 (5)	0.0061 (5)	−0.0030 (5)
C14	0.0156 (6)	0.0283 (7)	0.0228 (7)	−0.0028 (5)	0.0044 (5)	0.0045 (5)
C15	0.0205 (7)	0.0204 (6)	0.0220 (7)	−0.0001 (5)	0.0087 (6)	0.0039 (5)
C16	0.0171 (6)	0.0276 (7)	0.0178 (6)	−0.0063 (5)	0.0051 (5)	−0.0015 (5)
C17	0.0203 (7)	0.0358 (8)	0.0185 (7)	−0.0114 (6)	0.0082 (6)	−0.0080 (6)
C18	0.0284 (8)	0.0465 (9)	0.0350 (9)	−0.0084 (7)	0.0195 (7)	−0.0147 (7)
C19	0.0334 (9)	0.0564 (10)	0.0272 (8)	−0.0130 (8)	0.0160 (7)	−0.0021 (7)

Geometric parameters (Å, °)

C11—C13	1.7378 (13)	C8—C9	1.5291 (18)
C12—C15	1.7351 (13)	C8—H8A	0.9900
O1—C1	1.2071 (15)	C8—H8B	0.9900
O2—C1	1.3570 (15)	C9—H9A	0.9900
O2—C4	1.4562 (14)	C9—H9B	0.9900
O3—C3	1.3649 (15)	C10—C15	1.3904 (18)
O3—C16	1.3962 (16)	C10—C11	1.3943 (19)
O4—C16	1.1902 (16)	C11—C12	1.3870 (18)
C1—C2	1.4734 (17)	C11—H11	0.9500
C2—C3	1.3289 (18)	C12—C13	1.3807 (19)
C2—C10	1.4810 (17)	C12—H12	0.9500
C3—C4	1.5025 (17)	C13—C14	1.3789 (19)
C4—C5	1.5270 (18)	C14—C15	1.3933 (19)
C4—C9	1.5310 (17)	C14—H14	0.9500
C5—C6	1.5265 (18)	C16—C17	1.4827 (18)
C5—H5A	0.9900	C17—C18	1.337 (2)
C5—H5B	0.9900	C17—C19	1.493 (2)
C6—C7	1.5253 (19)	C18—H18A	0.9500
C6—H6A	0.9900	C18—H18B	0.9500
C6—H6B	0.9900	C19—H19A	0.9800
C7—C8	1.523 (2)	C19—H19B	0.9800
C7—H7A	0.9900	C19—H19C	0.9800
C7—H7B	0.9900		
C1—O2—C4	109.93 (9)	H8A—C8—H8B	107.9
C3—O3—C16	119.46 (10)	C8—C9—C4	111.66 (10)
O1—C1—O2	121.58 (11)	C8—C9—H9A	109.3
O1—C1—C2	128.67 (12)	C4—C9—H9A	109.3
O2—C1—C2	109.75 (10)	C8—C9—H9B	109.3
C3—C2—C1	105.91 (11)	C4—C9—H9B	109.3
C3—C2—C10	134.25 (12)	H9A—C9—H9B	108.0
C1—C2—C10	119.83 (11)	C15—C10—C11	117.74 (12)
C2—C3—O3	130.95 (12)	C15—C10—C2	122.54 (11)
C2—C3—C4	112.32 (11)	C11—C10—C2	119.49 (12)
O3—C3—C4	116.67 (11)	C12—C11—C10	121.34 (13)
O2—C4—C3	101.81 (10)	C12—C11—H11	119.3
O2—C4—C5	107.36 (10)	C10—C11—H11	119.3
C3—C4—C5	112.30 (10)	C13—C12—C11	119.05 (12)
O2—C4—C9	109.13 (10)	C13—C12—H12	120.5
C3—C4—C9	113.30 (10)	C11—C12—H12	120.5
C5—C4—C9	112.21 (10)	C14—C13—C12	121.58 (12)
C6—C5—C4	111.34 (10)	C14—C13—C11	118.88 (11)
C6—C5—H5A	109.4	C12—C13—C11	119.52 (10)
C4—C5—H5A	109.4	C13—C14—C15	118.31 (13)
C6—C5—H5B	109.4	C13—C14—H14	120.8
C4—C5—H5B	109.4	C15—C14—H14	120.8

H5A—C5—H5B	108.0	C10—C15—C14	121.92 (12)
C7—C6—C5	110.66 (11)	C10—C15—C12	119.98 (10)
C7—C6—H6A	109.5	C14—C15—C12	118.09 (10)
C5—C6—H6A	109.5	O4—C16—O3	122.02 (12)
C7—C6—H6B	109.5	O4—C16—C17	126.80 (12)
C5—C6—H6B	109.5	O3—C16—C17	111.16 (11)
H6A—C6—H6B	108.1	C18—C17—C16	120.88 (13)
C8—C7—C6	110.82 (11)	C18—C17—C19	124.49 (13)
C8—C7—H7A	109.5	C16—C17—C19	114.52 (13)
C6—C7—H7A	109.5	C17—C18—H18A	120.0
C8—C7—H7B	109.5	C17—C18—H18B	120.0
C6—C7—H7B	109.5	H18A—C18—H18B	120.0
H7A—C7—H7B	108.1	C17—C19—H19A	109.5
C7—C8—C9	111.89 (11)	C17—C19—H19B	109.5
C7—C8—H8A	109.2	H19A—C19—H19B	109.5
C9—C8—H8A	109.2	C17—C19—H19C	109.5
C7—C8—H8B	109.2	H19A—C19—H19C	109.5
C9—C8—H8B	109.2	H19B—C19—H19C	109.5
C4—O2—C1—O1	175.71 (11)	C7—C8—C9—C4	52.98 (15)
C4—O2—C1—C2	-4.42 (13)	O2—C4—C9—C8	67.09 (14)
O1—C1—C2—C3	-178.77 (13)	C3—C4—C9—C8	179.75 (11)
O2—C1—C2—C3	1.38 (14)	C5—C4—C9—C8	-51.77 (14)
O1—C1—C2—C10	1.1 (2)	C3—C2—C10—C15	-84.2 (2)
O2—C1—C2—C10	-178.79 (10)	C1—C2—C10—C15	95.97 (15)
C1—C2—C3—O3	-174.75 (13)	C3—C2—C10—C11	101.37 (18)
C10—C2—C3—O3	5.4 (3)	C1—C2—C10—C11	-78.41 (16)
C1—C2—C3—C4	2.16 (15)	C15—C10—C11—C12	-1.63 (19)
C10—C2—C3—C4	-177.64 (13)	C2—C10—C11—C12	173.02 (12)
C16—O3—C3—C2	-50.0 (2)	C10—C11—C12—C13	-0.5 (2)
C16—O3—C3—C4	133.19 (12)	C11—C12—C13—C14	2.3 (2)
C1—O2—C4—C3	5.30 (12)	C11—C12—C13—C11	-176.24 (10)
C1—O2—C4—C5	-112.83 (10)	C12—C13—C14—C15	-1.9 (2)
C1—O2—C4—C9	125.32 (10)	C11—C13—C14—C15	176.66 (10)
C2—C3—C4—O2	-4.59 (14)	C11—C10—C15—C14	2.05 (19)
O3—C3—C4—O2	172.81 (10)	C2—C10—C15—C14	-172.42 (12)
C2—C3—C4—C5	109.94 (13)	C11—C10—C15—C12	-178.86 (10)
O3—C3—C4—C5	-72.66 (14)	C2—C10—C15—C12	6.66 (18)
C2—C3—C4—C9	-121.62 (12)	C13—C14—C15—C10	-0.34 (19)
O3—C3—C4—C9	55.77 (15)	C13—C14—C15—C12	-179.44 (10)
O2—C4—C5—C6	-66.13 (13)	C3—O3—C16—O4	-14.1 (2)
C3—C4—C5—C6	-177.23 (10)	C3—O3—C16—C17	166.98 (11)
C9—C4—C5—C6	53.77 (14)	O4—C16—C17—C18	161.57 (15)
C4—C5—C6—C7	-56.48 (15)	O3—C16—C17—C18	-19.56 (18)
C5—C6—C7—C8	57.59 (15)	O4—C16—C17—C19	-14.7 (2)
C6—C7—C8—C9	-56.08 (15)	O3—C16—C17—C19	164.16 (12)

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>
C11—H11 $\cdots$ O1 ⁱ	0.95	2.52	3.2470 (17)	133
C18—H18 <i>B</i> $\cdots$ O1 ⁱⁱ	0.95	2.56	3.4486 (17)	156

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