



Crystal structure of methyl 1-methyl-2-oxospiro[indoline-3,2'-oxirane]-3'-carboxylate

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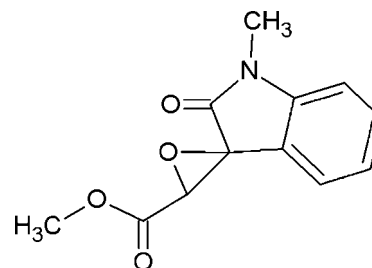
In the title compound, C₁₂H₁₁NO₄, the dihedral angle between the indole ring system (r.m.s. deviation = 0.019 Å) and the oxirane ring is 88.8 (2)°. The oxirane O atom and the bridging ester O atom are in an approximate syn conformation [O—C—C—O = −25.4 (3)°]. In the crystal, inversion dimers linked by pair of C—H···O hydrogen bonds generate R₂²(8) loops, where the C—H donor group forms part of the oxirane ring. A second C—H···O interaction arising from one of the C—H groups of the benzene ring links the dimers into [001] double chains.

Keywords: crystal structure; ester; spiro compound; indoline; oxirane; hydrogen bonding.

CCDC reference: 1056692

1. Related literature

For the bioactivity of indole derivatives, see: Di Fabio *et al.* (2007); Sharma & Tepe (2004). For a related structure, see: Savithri *et al.* (2015).



2. Experimental

2.1. Crystal data

C ₁₂ H ₁₁ NO ₄	$\gamma = 94.714 (3)^\circ$
$M_r = 233.22$	$V = 548.44 (5) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.1401 (4) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.7787 (4) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 9.0678 (4) \text{ \AA}$	$T = 293 \text{ K}$
$\alpha = 91.517 (3)^\circ$	$0.35 \times 0.30 \times 0.30 \text{ mm}$
$\beta = 104.227 (3)^\circ$	

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer	11311 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2004)	1927 independent reflections
$T_{\min} = 0.963$, $T_{\max} = 0.969$	1480 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.037$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.173$	
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
1927 reflections	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
158 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3···O3 ⁱ	0.95 (3)	2.52 (3)	3.414 (3)	157 (2)
C9—H9···O1 ⁱⁱ	0.93	2.43	3.335 (4)	163

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

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 for Windows (Farrugia, 2012); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7369).

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supporting information

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S1. Experimental

To a solution of the catalyst of diphenyl[(*R*)-2-pyrrolidinyl]methanol (0.15mmol) and trans- α -ylideneoxindoles 1 (0.5 mmol) in nHexane for HPLC grade (2.7ml) was added TBHP (5.5M in decane solution, 0.6mol) at room temperature (25°C). The resultant heterogeneous mixture was maintained under stirring until the reaction completion (TLC nHexane/EtOAc). After wards, the crude reaction mixture was purified by flash chromatography on silica gel (nHexane/EtOAc) to furnish the epoxy oxindoles trans-2 and cis-3. Colourless blocks were obtained by slow evaporation of a solution of the title compound in ethyl acetate at room temperature.

S2. Refinement

All H atoms were fixed geometrically and allowed to ride on their parent C atoms, with C-H distances fixed in the range 0.93-0.98 Å with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H 1.2 $U_{eq}(C)$ for other H atoms.

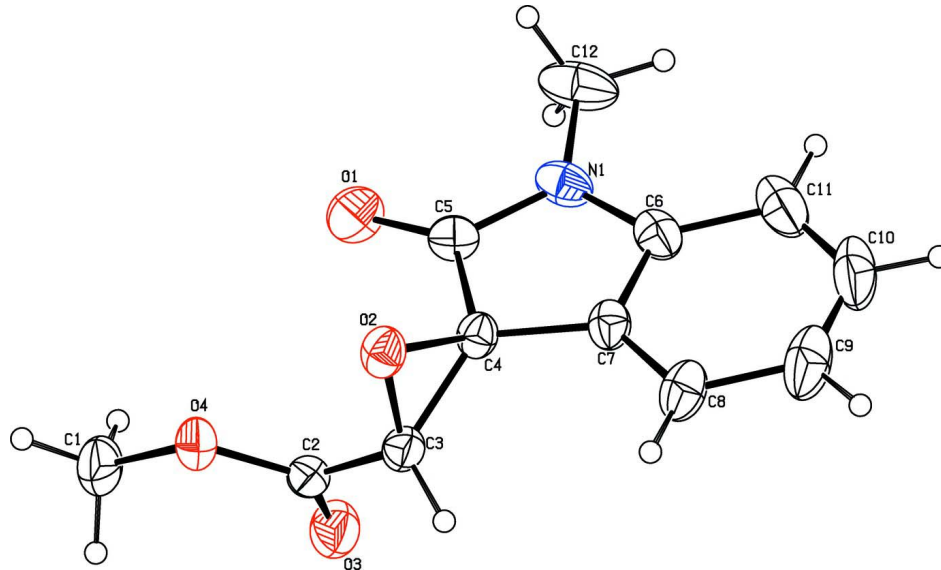


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

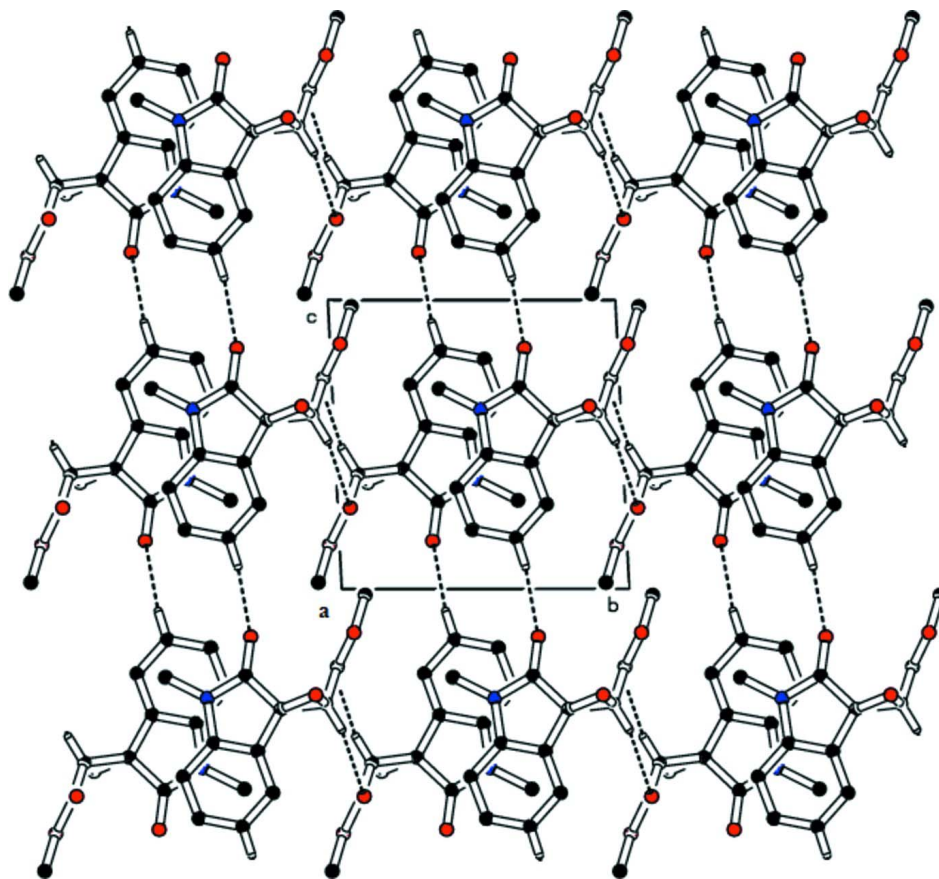


Figure 2

The molecular packing as viewed along the *a* axis. Dashed lines shows the C—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity.

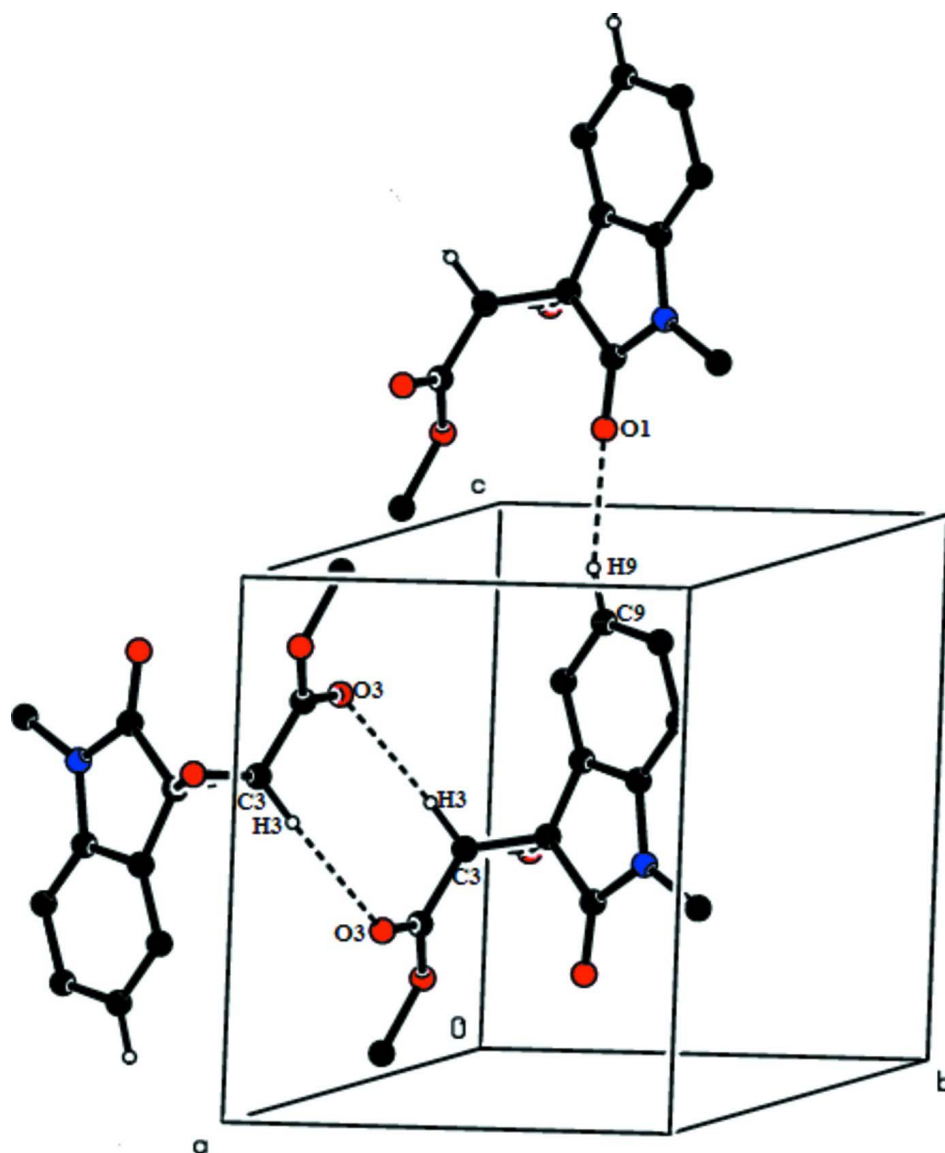


Figure 3

A partial view of the hydrogen-bond interactions C9—H9...O1 and C3—H3...O3 along *a* axis.

Methyl 1-methyl-2-oxospiro[indoline-3,2'-oxirane]-3'-carboxylate

Crystal data

$C_{12}H_{11}NO_4$

$M_r = 233.22$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.1401(4)\ \text{\AA}$

$b = 8.7787(4)\ \text{\AA}$

$c = 9.0678(4)\ \text{\AA}$

$\alpha = 91.517(3)^\circ$

$\beta = 104.227(3)^\circ$

$\gamma = 94.714(3)^\circ$

$V = 548.44(5)\ \text{\AA}^3$

$Z = 2$

$F(000) = 244$

$D_x = 1.412\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1927 reflections

$\theta = 2.3\text{--}25.0^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.35 \times 0.30 \times 0.30\ \text{mm}$

Data collection

Bruker Kappa APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω and φ scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2004)
 $T_{\min} = 0.963$, $T_{\max} = 0.969$

11311 measured reflections
 1927 independent reflections
 1480 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.3^\circ$
 $h = -8 \rightarrow 8$
 $k = -10 \rightarrow 10$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.173$
 $S = 1.06$
 1927 reflections
 158 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.102P)^2 + 0.172P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.30 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.0254 (2)	0.10821 (18)	0.36778 (18)	0.0462 (5)
O4	0.1745 (3)	-0.03538 (19)	0.15149 (19)	0.0536 (5)
O3	0.4764 (3)	0.0432 (2)	0.2809 (2)	0.0632 (6)
O1	0.1739 (3)	0.3246 (2)	0.1663 (2)	0.0643 (6)
N1	0.2372 (3)	0.4866 (2)	0.3794 (3)	0.0504 (6)
C7	0.2040 (3)	0.3195 (3)	0.5623 (3)	0.0425 (6)
C2	0.3047 (4)	0.0282 (2)	0.2698 (3)	0.0429 (6)
C4	0.1736 (3)	0.2320 (2)	0.4166 (2)	0.0379 (5)
C3	0.2223 (4)	0.0750 (3)	0.3974 (3)	0.0428 (6)
C6	0.2456 (3)	0.4711 (3)	0.5343 (3)	0.0486 (6)
C5	0.1928 (3)	0.3492 (3)	0.3012 (3)	0.0438 (6)
C8	0.2075 (4)	0.2738 (4)	0.7065 (3)	0.0566 (7)
H8	0.1811	0.1715	0.7247	0.068*
C11	0.2901 (4)	0.5825 (4)	0.6509 (4)	0.0717 (10)
H11	0.3174	0.6849	0.6334	0.086*

C1	0.2456 (5)	-0.0803 (4)	0.0221 (3)	0.0692 (9)
H1A	0.1393	-0.1255	-0.0577	0.104*
H1B	0.3391	-0.1534	0.0521	0.104*
H1C	0.3056	0.0081	-0.0141	0.104*
C12	0.2681 (5)	0.6277 (3)	0.3066 (5)	0.0774 (10)
H12A	0.2979	0.7114	0.3813	0.116*
H12B	0.1528	0.6440	0.2303	0.116*
H12C	0.3742	0.6214	0.2597	0.116*
C10	0.2921 (5)	0.5344 (5)	0.7951 (4)	0.0845 (12)
H10	0.3223	0.6068	0.8761	0.101*
C9	0.2516 (5)	0.3848 (5)	0.8237 (4)	0.0796 (11)
H9	0.2537	0.3576	0.9225	0.096*
H3	0.273 (4)	0.024 (3)	0.488 (3)	0.052 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0454 (10)	0.0440 (9)	0.0450 (9)	-0.0086 (7)	0.0076 (7)	0.0005 (7)
O4	0.0565 (11)	0.0523 (11)	0.0456 (10)	0.0033 (8)	0.0026 (8)	-0.0115 (8)
O3	0.0524 (13)	0.0730 (13)	0.0597 (12)	0.0065 (9)	0.0068 (9)	-0.0112 (9)
O1	0.0859 (15)	0.0673 (12)	0.0455 (11)	0.0096 (10)	0.0247 (10)	0.0167 (9)
N1	0.0464 (12)	0.0339 (11)	0.0738 (15)	0.0021 (8)	0.0206 (10)	0.0087 (9)
C7	0.0352 (12)	0.0495 (14)	0.0418 (13)	0.0004 (10)	0.0099 (9)	-0.0061 (10)
C2	0.0477 (15)	0.0335 (12)	0.0425 (13)	0.0041 (10)	0.0020 (10)	0.0010 (9)
C4	0.0380 (12)	0.0387 (12)	0.0351 (11)	-0.0034 (9)	0.0078 (9)	0.0011 (9)
C3	0.0477 (14)	0.0373 (12)	0.0382 (13)	-0.0010 (10)	0.0024 (10)	0.0032 (10)
C6	0.0333 (13)	0.0470 (14)	0.0650 (17)	0.0033 (10)	0.0125 (11)	-0.0104 (11)
C5	0.0425 (13)	0.0441 (13)	0.0476 (14)	0.0047 (10)	0.0154 (10)	0.0087 (10)
C8	0.0495 (15)	0.0805 (19)	0.0392 (14)	-0.0009 (13)	0.0128 (11)	-0.0032 (12)
C11	0.0491 (17)	0.0595 (18)	0.100 (3)	0.0038 (13)	0.0116 (16)	-0.0328 (17)
C1	0.077 (2)	0.082 (2)	0.0455 (15)	0.0188 (16)	0.0083 (13)	-0.0170 (14)
C12	0.0574 (18)	0.0453 (16)	0.137 (3)	0.0067 (13)	0.0348 (18)	0.0312 (17)
C10	0.061 (2)	0.110 (3)	0.074 (2)	0.0127 (19)	0.0073 (16)	-0.052 (2)
C9	0.062 (2)	0.128 (3)	0.0476 (17)	0.0067 (19)	0.0138 (14)	-0.0251 (18)

Geometric parameters (Å, °)

O2—C3	1.421 (3)	C3—H3	0.95 (3)
O2—C4	1.433 (3)	C6—C11	1.380 (4)
O4—C2	1.311 (3)	C8—C9	1.382 (4)
O4—C1	1.446 (3)	C8—H8	0.9300
O3—C2	1.201 (3)	C11—C10	1.382 (5)
O1—C5	1.209 (3)	C11—H11	0.9300
N1—C5	1.356 (3)	C1—H1A	0.9600
N1—C6	1.402 (3)	C1—H1B	0.9600
N1—C12	1.446 (3)	C1—H1C	0.9600
C7—C8	1.373 (3)	C12—H12A	0.9600
C7—C6	1.384 (4)	C12—H12B	0.9600

C7—C4	1.470 (3)	C12—H12C	0.9600
C2—C3	1.484 (4)	C10—C9	1.367 (5)
C4—C3	1.466 (3)	C10—H10	0.9300
C4—C5	1.509 (3)	C9—H9	0.9300
C3—O2—C4	61.80 (14)	O1—C5—C4	126.6 (2)
C2—O4—C1	116.2 (2)	N1—C5—C4	106.3 (2)
C5—N1—C6	111.2 (2)	C7—C8—C9	117.8 (3)
C5—N1—C12	122.4 (3)	C7—C8—H8	121.1
C6—N1—C12	126.4 (2)	C9—C8—H8	121.1
C8—C7—C6	121.6 (2)	C6—C11—C10	116.7 (3)
C8—C7—C4	131.6 (2)	C6—C11—H11	121.7
C6—C7—C4	106.7 (2)	C10—C11—H11	121.7
O3—C2—O4	125.0 (2)	O4—C1—H1A	109.5
O3—C2—C3	121.3 (2)	O4—C1—H1B	109.5
O4—C2—C3	113.7 (2)	H1A—C1—H1B	109.5
O2—C4—C3	58.71 (14)	O4—C1—H1C	109.5
O2—C4—C7	123.07 (19)	H1A—C1—H1C	109.5
C3—C4—C7	125.9 (2)	H1B—C1—H1C	109.5
O2—C4—C5	116.78 (18)	N1—C12—H12A	109.5
C3—C4—C5	121.0 (2)	N1—C12—H12B	109.5
C7—C4—C5	105.51 (19)	H12A—C12—H12B	109.5
O2—C3—C4	59.48 (14)	N1—C12—H12C	109.5
O2—C3—C2	119.95 (19)	H12A—C12—H12C	109.5
C4—C3—C2	121.3 (2)	H12B—C12—H12C	109.5
O2—C3—H3	117.1 (15)	C9—C10—C11	122.8 (3)
C4—C3—H3	116.4 (16)	C9—C10—H10	118.6
C2—C3—H3	112.8 (15)	C11—C10—H10	118.6
C11—C6—C7	120.9 (3)	C10—C9—C8	120.3 (3)
C11—C6—N1	128.8 (3)	C10—C9—H9	119.9
C7—C6—N1	110.3 (2)	C8—C9—H9	119.9
O1—C5—N1	127.2 (2)		
C1—O4—C2—O3	-4.4 (4)	C4—C7—C6—N1	2.0 (3)
C1—O4—C2—C3	178.6 (2)	C5—N1—C6—C11	178.4 (2)
C3—O2—C4—C7	-115.0 (2)	C12—N1—C6—C11	-2.3 (4)
C3—O2—C4—C5	111.7 (2)	C5—N1—C6—C7	-0.6 (3)
C8—C7—C4—O2	44.0 (4)	C12—N1—C6—C7	178.7 (2)
C6—C7—C4—O2	-140.1 (2)	C6—N1—C5—O1	179.6 (2)
C8—C7—C4—C3	-28.9 (4)	C12—N1—C5—O1	0.2 (4)
C6—C7—C4—C3	147.0 (2)	C6—N1—C5—C4	-1.0 (3)
C8—C7—C4—C5	-178.4 (2)	C12—N1—C5—C4	179.7 (2)
C6—C7—C4—C5	-2.5 (2)	O2—C4—C5—O1	-37.7 (3)
C4—O2—C3—C2	-110.9 (2)	C3—C4—C5—O1	30.3 (4)
C7—C4—C3—O2	110.4 (2)	C7—C4—C5—O1	-178.4 (2)
C5—C4—C3—O2	-104.4 (2)	O2—C4—C5—N1	142.9 (2)
O2—C4—C3—C2	108.6 (2)	C3—C4—C5—N1	-149.1 (2)
C7—C4—C3—C2	-141.0 (2)	C7—C4—C5—N1	2.2 (2)

C5—C4—C3—C2	4.2 (3)	C6—C7—C8—C9	0.9 (4)
O3—C2—C3—O2	157.5 (2)	C4—C7—C8—C9	176.2 (3)
O4—C2—C3—O2	-25.4 (3)	C7—C6—C11—C10	0.5 (4)
O3—C2—C3—C4	87.1 (3)	N1—C6—C11—C10	-178.5 (2)
O4—C2—C3—C4	-95.8 (2)	C6—C11—C10—C9	-0.3 (5)
C8—C7—C6—C11	-0.8 (4)	C11—C10—C9—C8	0.5 (5)
C4—C7—C6—C11	-177.1 (2)	C7—C8—C9—C10	-0.7 (4)
C8—C7—C6—N1	178.4 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...O3 ⁱ	0.95 (3)	2.52 (3)	3.414 (3)	157 (2)
C9—H9...O1 ⁱⁱ	0.93	2.43	3.335 (4)	163

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