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Methyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-acetate 1,1-dioxide

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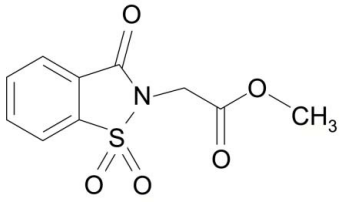
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 15.9.

The title molecule, $C_{10}H_9NO_5S$, is composed of two essentially planar units with a dihedral angle of 89.16 (6)° between them. In the crystal structure, there are weak intermolecular C—H···O interactions resulting in dimeric pairs of molecules about inversion centres and chains of molecules extended along the a and c axes, thus stabilizing the structure. In addition, benzothiazole rings lying parallel to each other with centroid–centroid distances of 3.679 (2) and 3.999 (2) Å indicate the existence of π–π stacking interactions.

Related literature

For related literature, see: Kapui *et al.* (2003); Masashi *et al.* (1999); Manjarrez *et al.* (1996); Siddiqui, Ahmad, Khan, Siddiqui & Parvez (2007); Siddiqui, Ahmad, Khan, Siddiqui & Weaver (2007); Siddiqui, Ahmad, Siddiqui *et al.* (2007); Siddiqui *et al.* (2008); Xu *et al.* (2005, 2006).



Experimental

Crystal data

$C_{10}H_9NO_5S$
M_r = 255.24
Triclinic, $P\overline{1}$
a = 7.765 (3) Å
b = 8.496 (3) Å
c = 8.776 (4) Å
α = 104.39 (2)°
β = 100.58 (2)°
γ = 94.30 (2)°
V = 546.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 173 (2) K
0.16 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.953, T_{max} = 0.976
4654 measured reflections
2468 independent reflections
2040 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.03
2468 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O5^{i}	0.95	2.53	3.435 (3)	160
C4—H4···O4^{ii}	0.95	2.54	3.209 (3)	128
C8—H8A···O2^{iii}	0.99	2.49	3.435 (3)	159
C10—H10C···O1^{iv}	0.98	2.47	3.431 (3)	167

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2607).

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supporting information

Acta Cryst. (2008). E**64**, o859 [doi:10.1107/S1600536808009951]

Methyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-acetate 1,1-dioxide

Waseeq Ahmad Siddiqui, Saeed Ahmad, Hamid Latif Siddiqui, Masood Parvez and Rehana Rashid

S1. Comment

Saccharin derivatives are considered to be the most potent orally active human leucocyte elastase (HLE) inhibitors for the treatment of chronic obstructive pulmonary disease (COPD), acute respiratory distress syndrome (ARDS), cystic fibrosis, asthma and other inflammatory diseases (Kapui *et al.*, 2003). Various biologically important saccharin skeletons and their *N*-alkyl derivatives were efficiently prepared (Xu *et al.*, 2006) by chromium oxide-catalyzed oxidation of *N*-alkyl(*o*-methyl)arenesulfonamides in acetonitrile besides the already developed methodology utilizing irradiation techniques for similar type of conversions (Masashi *et al.*, 1999). In continuation to our research on benzene, 1,2-benzothiazine 1,1-dioxide and saccharin derivatives (Siddiqui *et al.*, 2008; Siddiqui, Ahmad, Khan, Siddiqui & Weaver, 2007), we report herein the crystal structure of the title compound, (I).

The structure of (I) is composed of an essentially planar moiety, S1/N1/O1/C1—C7 with maximum deviations from the least-square planes being: O1 = -0.0540 (12) and N1 = 0.0540 (13) Å, and an approximately planar moiety C8—C10/O4/O5 with maximum deviation of 0.0766 (13) Å for C8. The two moieties are oriented with a dihedral angle of 89.16 (6)° between their least-squares planes. The structure is stabilized by four rather weak intermolecular interactions of the type C—H···O (Fig. 2 and Table 1). Of these interactions, C4—H4···O5 and C8—H8A···O2 H-bonds result in dimeric pairs of (I) while C10—H10···O1 and C2—H2···O5 result in chains of molecules extended along the a- and c-axes, respectively. The benzothiazole rings in (I) lie parallel to each other about the origin with the shortest distance between the centroids of the benzene rings of the adjacent molecules is 3.679 (2) Å which indicates the existence of π-π stacking interactions. The thiazoline rings located about inversion centers in the middle of the *b* axis (at 0, 1/2, 0) also show π-π interaction with centroids of these rings separated by 3.999 (2) Å (Fig. 3). The molecular dimensions in (I) are in agreement with the corresponding dimensions reported in similar structures (Xu *et al.*, 2005; Siddiqui, Ahmad, Khan, Siddiqui & Parvez, 2007; Siddiqui, Ahmad, Siddiqui *et al.*, 2007; Siddiqui *et al.*, 2008).

S2. Experimental

The compound (I) was prepared following the prcedures reported earlier (Manjarrez *et al.*, 1996). Crystals suitable for X-ray crystallography were grown from a solution of CH_3OH by slow evaporation at 313 K.

S3. Refinement

H-atoms were included in the refinements at geometrically idealized positions with aryl, methylene and methyl C—H distances 0.95, 0.99 and 0.98 Å, respectively, and U_{iso} = 1.2 times U_{eq} of the atoms to which they were bonded. The final difference map was free of any chemically significant features.

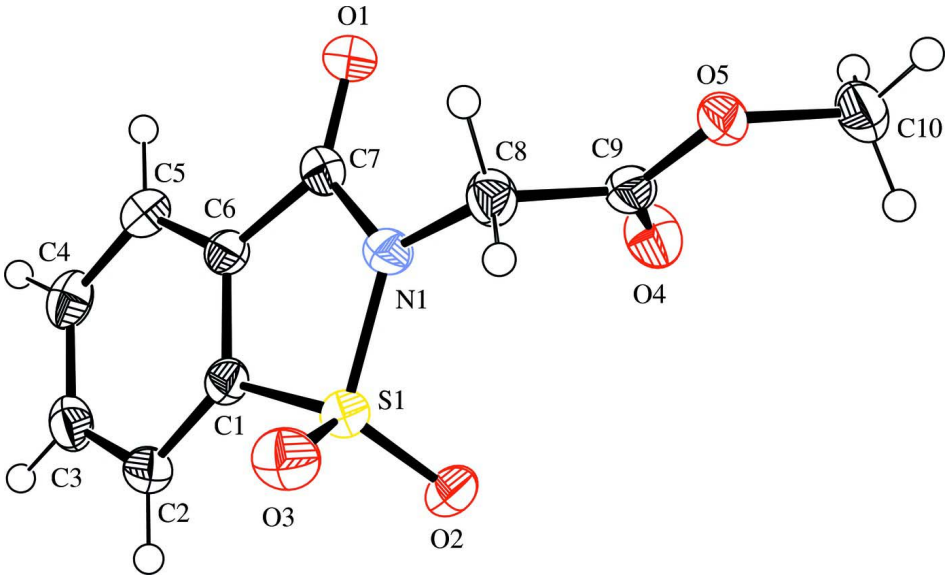

Figure 1

ORTEP-3 (Farrugia, 1997) drawing of (I) with displacement ellipsoids plotted at 50% probability level.

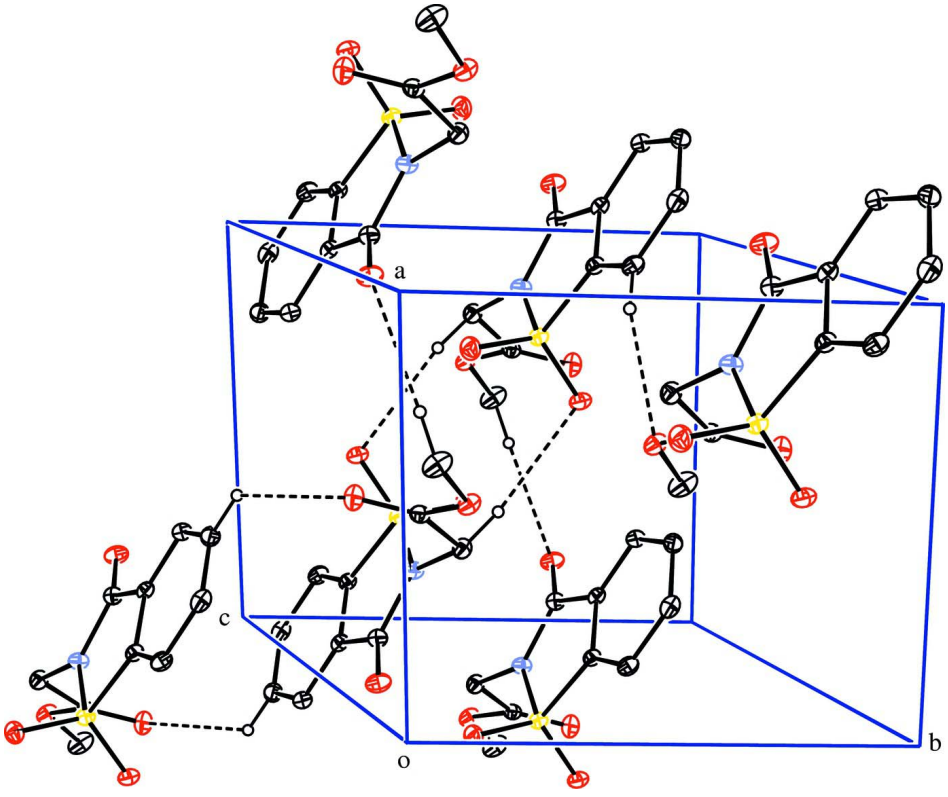

Figure 2

Unit cell packing of (I) showing C—H···O interactions represented by dashed lines; H-atoms not involved in H-bonds have been omitted.

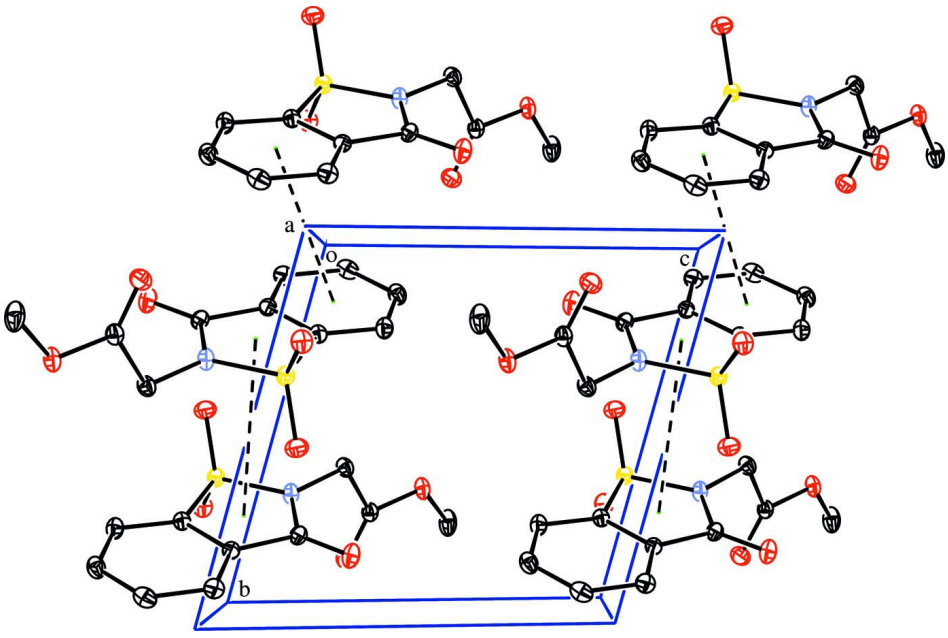

Figure 3

Unit cell packing of (I) showing π-π stacking interactions represented by dashed lines; H-atoms have been omitted for clarity.

Methyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-acetate 1,1-dioxide

Crystal data

$C_{10}H_9NO_5S$
M_r = 255.24
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.765 (3) Å
b = 8.496 (3) Å
c = 8.776 (4) Å
α = 104.39 (2)°
β = 100.58 (2)°
γ = 94.30 (2)°
V = 546.8 (4) Å^3
Z = 2
F(000) = 264
D_x = 1.550 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4654 reflections
θ = 3.9–27.4°
μ = 0.31 mm^{-1}
T = 173 K
Block, colourless
0.16 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.953, T_{max} = 0.976
4654 measured reflections
2468 independent reflections
2040 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.4°, θ_{min} = 3.9°
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.03$
2468 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.29P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.25284 (6)	0.37128 (6)	1.06522 (6)	0.02602 (16)
O1	−0.07412 (18)	0.18372 (19)	0.67891 (17)	0.0354 (3)
O2	0.40672 (17)	0.29152 (17)	1.08988 (17)	0.0320 (3)
O3	0.2727 (2)	0.54422 (17)	1.13620 (18)	0.0372 (4)
O4	0.3882 (2)	0.14876 (17)	0.70088 (18)	0.0369 (4)
O5	0.41016 (18)	0.33528 (17)	0.56027 (16)	0.0312 (3)
N1	0.1635 (2)	0.3311 (2)	0.87005 (18)	0.0275 (4)
C1	0.0687 (2)	0.2676 (2)	1.1032 (2)	0.0243 (4)
C2	0.0451 (3)	0.2516 (2)	1.2517 (2)	0.0303 (4)
H2	0.1321	0.2987	1.3471	0.036*
C3	−0.1118 (3)	0.1633 (3)	1.2543 (3)	0.0325 (4)
H3	−0.1321	0.1482	1.3535	0.039*
C4	−0.2397 (3)	0.0968 (2)	1.1148 (3)	0.0329 (5)
H4	−0.3467	0.0384	1.1202	0.040*
C5	−0.2128 (3)	0.1147 (2)	0.9669 (2)	0.0297 (4)
H5	−0.3005	0.0698	0.8716	0.036*
C6	−0.0558 (2)	0.1990 (2)	0.9619 (2)	0.0245 (4)
C7	−0.0003 (2)	0.2326 (2)	0.8179 (2)	0.0263 (4)
C8	0.2499 (3)	0.3960 (2)	0.7616 (2)	0.0292 (4)
H8A	0.3291	0.4972	0.8238	0.035*
H8B	0.1593	0.4256	0.6819	0.035*
C9	0.3566 (2)	0.2757 (2)	0.6733 (2)	0.0268 (4)
C10	0.5248 (3)	0.2391 (3)	0.4726 (3)	0.0435 (6)
H10A	0.5553	0.2910	0.3914	0.052*
H10B	0.4638	0.1284	0.4195	0.052*

H10C	0.6329	0.2329	0.5475	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0241 (3)	0.0292 (3)	0.0245 (3)	0.00447 (18)	0.00385 (18)	0.00737 (18)
O1	0.0311 (7)	0.0495 (9)	0.0236 (7)	0.0080 (6)	0.0024 (6)	0.0076 (6)
O2	0.0225 (7)	0.0398 (8)	0.0339 (8)	0.0068 (6)	0.0027 (6)	0.0117 (6)
O3	0.0426 (9)	0.0266 (7)	0.0379 (8)	0.0026 (6)	0.0035 (7)	0.0045 (6)
O4	0.0472 (9)	0.0316 (8)	0.0378 (8)	0.0101 (7)	0.0158 (7)	0.0136 (6)
O5	0.0308 (7)	0.0441 (8)	0.0252 (7)	0.0126 (6)	0.0102 (6)	0.0158 (6)
N1	0.0242 (8)	0.0376 (9)	0.0217 (8)	0.0022 (7)	0.0048 (6)	0.0102 (7)
C1	0.0226 (9)	0.0270 (9)	0.0251 (9)	0.0069 (7)	0.0061 (7)	0.0083 (7)
C2	0.0307 (10)	0.0375 (11)	0.0241 (10)	0.0094 (8)	0.0053 (8)	0.0095 (8)
C3	0.0377 (11)	0.0375 (11)	0.0309 (11)	0.0155 (9)	0.0161 (9)	0.0151 (8)
C4	0.0269 (10)	0.0328 (10)	0.0452 (12)	0.0065 (8)	0.0137 (9)	0.0164 (9)
C5	0.0245 (9)	0.0314 (10)	0.0328 (10)	0.0046 (8)	0.0035 (8)	0.0095 (8)
C6	0.0252 (9)	0.0248 (9)	0.0240 (9)	0.0086 (7)	0.0042 (7)	0.0066 (7)
C7	0.0261 (9)	0.0288 (9)	0.0237 (10)	0.0051 (7)	0.0073 (7)	0.0042 (7)
C8	0.0305 (10)	0.0329 (10)	0.0297 (10)	0.0075 (8)	0.0115 (8)	0.0135 (8)
C9	0.0230 (9)	0.0325 (10)	0.0243 (9)	0.0000 (7)	0.0032 (7)	0.0090 (8)
C10	0.0436 (13)	0.0656 (15)	0.0302 (11)	0.0250 (11)	0.0173 (10)	0.0169 (10)

Geometric parameters (Å, º)

S1—O2	1.4258 (15)	C3—C4	1.389 (3)
S1—O3	1.4306 (15)	C3—H3	0.9500
S1—N1	1.6640 (18)	C4—C5	1.395 (3)
S1—C1	1.7504 (19)	C4—H4	0.9500
O1—C7	1.202 (2)	C5—C6	1.380 (3)
O4—C9	1.196 (2)	C5—H5	0.9500
O5—C9	1.335 (2)	C6—C7	1.492 (3)
O5—C10	1.449 (2)	C8—C9	1.517 (3)
N1—C7	1.402 (3)	C8—H8A	0.9900
N1—C8	1.449 (2)	C8—H8B	0.9900
C1—C2	1.387 (3)	C10—H10A	0.9800
C1—C6	1.388 (3)	C10—H10B	0.9800
C2—C3	1.389 (3)	C10—H10C	0.9800
C2—H2	0.9500		
O2—S1—O3	116.71 (9)	C6—C5—H5	120.7
O2—S1—N1	110.73 (9)	C4—C5—H5	120.7
O3—S1—N1	109.35 (9)	C5—C6—C1	119.81 (18)
O2—S1—C1	112.26 (9)	C5—C6—C7	127.38 (17)
O3—S1—C1	112.83 (9)	C1—C6—C7	112.78 (17)
N1—S1—C1	92.26 (9)	O1—C7—N1	123.33 (18)
C9—O5—C10	115.33 (16)	O1—C7—C6	128.71 (18)
C7—N1—C8	122.31 (16)	N1—C7—C6	107.93 (16)

C7—N1—S1	116.00 (13)	N1—C8—C9	112.82 (16)
C8—N1—S1	121.68 (13)	N1—C8—H8A	109.0
C2—C1—C6	122.73 (18)	C9—C8—H8A	109.0
C2—C1—S1	126.37 (15)	N1—C8—H8B	109.0
C6—C1—S1	110.90 (14)	C9—C8—H8B	109.0
C1—C2—C3	116.80 (19)	H8A—C8—H8B	107.8
C1—C2—H2	121.6	O4—C9—O5	125.61 (18)
C3—C2—H2	121.6	O4—C9—C8	125.59 (18)
C2—C3—C4	121.34 (19)	O5—C9—C8	108.80 (16)
C2—C3—H3	119.3	O5—C10—H10A	109.5
C4—C3—H3	119.3	O5—C10—H10B	109.5
C3—C4—C5	120.73 (19)	H10A—C10—H10B	109.5
C3—C4—H4	119.6	O5—C10—H10C	109.5
C5—C4—H4	119.6	H10A—C10—H10C	109.5
C6—C5—C4	118.57 (18)	H10B—C10—H10C	109.5

O2—S1—N1—C7	111.37 (15)	C2—C1—C6—C5	1.8 (3)
O3—S1—N1—C7	−118.66 (15)	S1—C1—C6—C5	−178.65 (14)
C1—S1—N1—C7	−3.48 (15)	C2—C1—C6—C7	−179.84 (17)
O2—S1—N1—C8	−69.24 (17)	S1—C1—C6—C7	−0.3 (2)
O3—S1—N1—C8	60.73 (17)	C8—N1—C7—O1	6.2 (3)
C1—S1—N1—C8	175.91 (15)	S1—N1—C7—O1	−174.37 (15)
O2—S1—C1—C2	68.07 (19)	C8—N1—C7—C6	−175.64 (16)
O3—S1—C1—C2	−66.31 (19)	S1—N1—C7—C6	3.7 (2)
N1—S1—C1—C2	−178.43 (18)	C5—C6—C7—O1	−5.9 (3)
O2—S1—C1—C6	−111.44 (14)	C1—C6—C7—O1	175.94 (19)
O3—S1—C1—C6	114.19 (14)	C5—C6—C7—N1	176.14 (18)
N1—S1—C1—C6	2.07 (14)	C1—C6—C7—N1	−2.0 (2)
C6—C1—C2—C3	−0.4 (3)	C7—N1—C8—C9	−84.4 (2)
S1—C1—C2—C3	−179.86 (15)	S1—N1—C8—C9	96.29 (18)
C1—C2—C3—C4	−1.0 (3)	C10—O5—C9—O4	−3.8 (3)
C2—C3—C4—C5	1.0 (3)	C10—O5—C9—C8	175.82 (16)
C3—C4—C5—C6	0.4 (3)	N1—C8—C9—O4	−9.1 (3)
C4—C5—C6—C1	−1.8 (3)	N1—C8—C9—O5	171.21 (15)
C4—C5—C6—C7	−179.84 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O5^{i}	0.95	2.53	3.435 (3)	160
C4—H4···O4^{ii}	0.95	2.54	3.209 (3)	128
C8—H8*A*···O2^{iii}	0.99	2.49	3.435 (3)	159
C10—H10*C*···O1^{iv}	0.98	2.47	3.431 (3)	167

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