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(5*E*)-5-(4-Methoxybenzylidene)-2-(piperidin-1-yl)-1,3-thiazol-4(5*H*)-one

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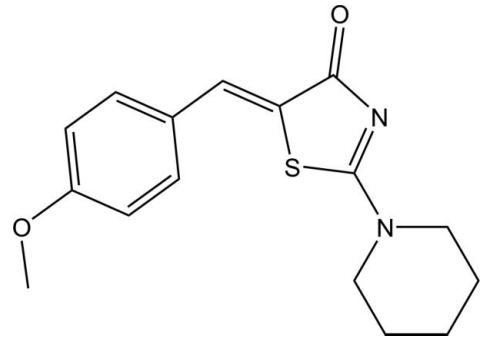
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.119; data-to-parameter ratio = 28.4.

In the title compound, $C_{16}H_{18}N_2O_2S$, the piperidine ring adopts a chair conformation. The central 4-thiazolidinone ring makes dihedral angles of 12.01 (7) and 51.42 (9)°, respectively, with the benzene ring and the least-squares plane of the piperidine ring. An intramolecular C—H···S hydrogen bond stabilizes the molecular structure and generates an S(6) ring motif. In the crystal, molecules are linked into a tape along the c axis by intermolecular C—H···O hydrogen bonds.

Related literature

For general background to the title compound, see: Lesyk & Zimenkovsky (2004); Lesyk *et al.* (2007); Havrylyuk *et al.* (2009); Ahn *et al.* (2006); Park *et al.* (2008); Geronikaki *et al.* (2008); Zimenkovsky *et al.* (2005). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{18}N_2O_2S$ M_r = 302.38
Monoclinic, $P2_1/c$
a = 8.5811 (3) Å
b = 16.5165 (6) Å
c = 12.4930 (4) Å
β = 121.518 (2)°
V = 1509.42 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 297 K
0.61 × 0.26 × 0.23 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.877, T_{max} = 0.950
20021 measured reflections
5432 independent reflections
4148 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.02
5432 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{i}	0.93	2.48	3.3048 (16)	147
C5—H5A···S1	0.93	2.58	3.2809 (15)	132
C16—H16A···O1^{ii}	0.96	2.48	3.421 (3)	167

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2743).

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supporting information

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(5*E*)-5-(4-Methoxybenzylidene)-2-(piperidin-1-yl)-1,3-thiazol-4(5*H*)-one

Hoong-Kun Fun, Chin Sing Yeap, Prajwal L. Lobo, D. Jagadeesh Prasad and Boja Poojary

S1. Comment

4-Thiazolidinone ring system is a core structure in various synthetic compounds which display a broad spectrum of biological activities (Lesyk & Zimenkovsky, 2004) including an anticancer effect (Lesyk *et al.*, 2007; Havrylyuk *et al.*, 2009). Mechanisms of 4-thiazolidinones and related heterocycles antitumor activity may be associated with the affinity to anticancer bio-targets, such as phosphatase of a regenerating liver (PRL-3) (Ahn *et al.*, 2006; Park *et al.*, 2008) and non-membrane protein tyrosine phosphatase (SHP-2) (Geronikaki *et al.*, 2008). 5-Arylidene derivatives were previously shown as the most active group of compounds with the anticancer activity among a large pool of 4-azolidone derivatives and analogs (Zimenkovsky *et al.*, 2005). This prompted us to synthesize title compound (I).

The central 4-thiazolidinone ring makes dihedral angles of 12.01 (7) and 51.42 (9)°, respectively, with the benzene ring and the least-squares plane of piperidine ring. The piperidine ring adopts a chair conformation. An intramolecular C5—H5A···S1 hydrogen bond (Table 1) stabilizes the molecular structure and generates an *S*(6) ring motif (Fig. 1; Bernstein *et al.*, 1995). In the crystal structure, the molecules are linked into a tape along the *c* axis by intermolecular C16—H16A···O1 and C2—H2A···O1 hydrogen bonds (Table 1 and Fig. 2).

S2. Experimental

An equimolar mixture of 2-(piperidin-1-yl)-1,3-thiazol-4(5*H*)-one, anisaldehyde and sodium acetate in acetic acid was refluxed for 2 hrs. The product formed was filtered, washed, dried and re-crystallized from ethanol.

S3. Refinement

All hydrogen atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). A rotating-group model were applied for methyl group.

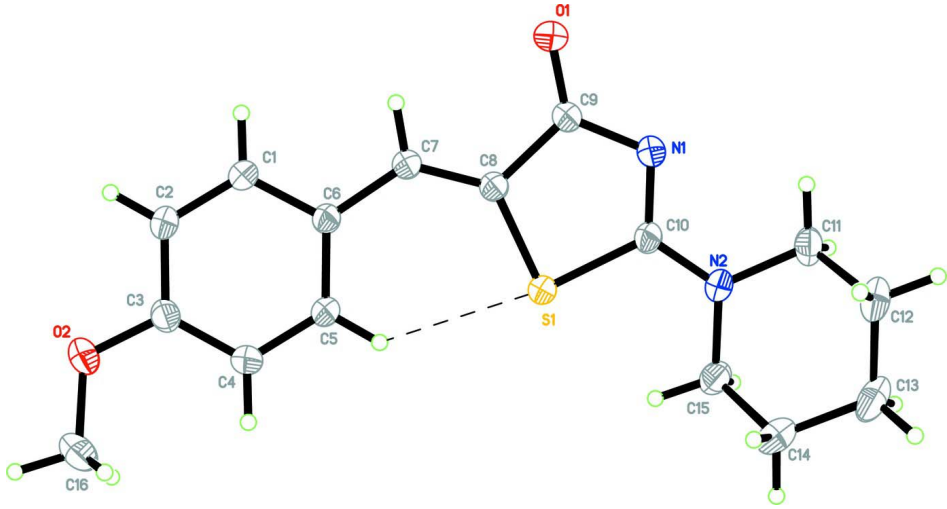

Figure 1

The molecular structure of the title compound, with atom labels and 30% probability ellipsoids for non-H atoms. Hydrogen bonds (dashed lines) are shown.

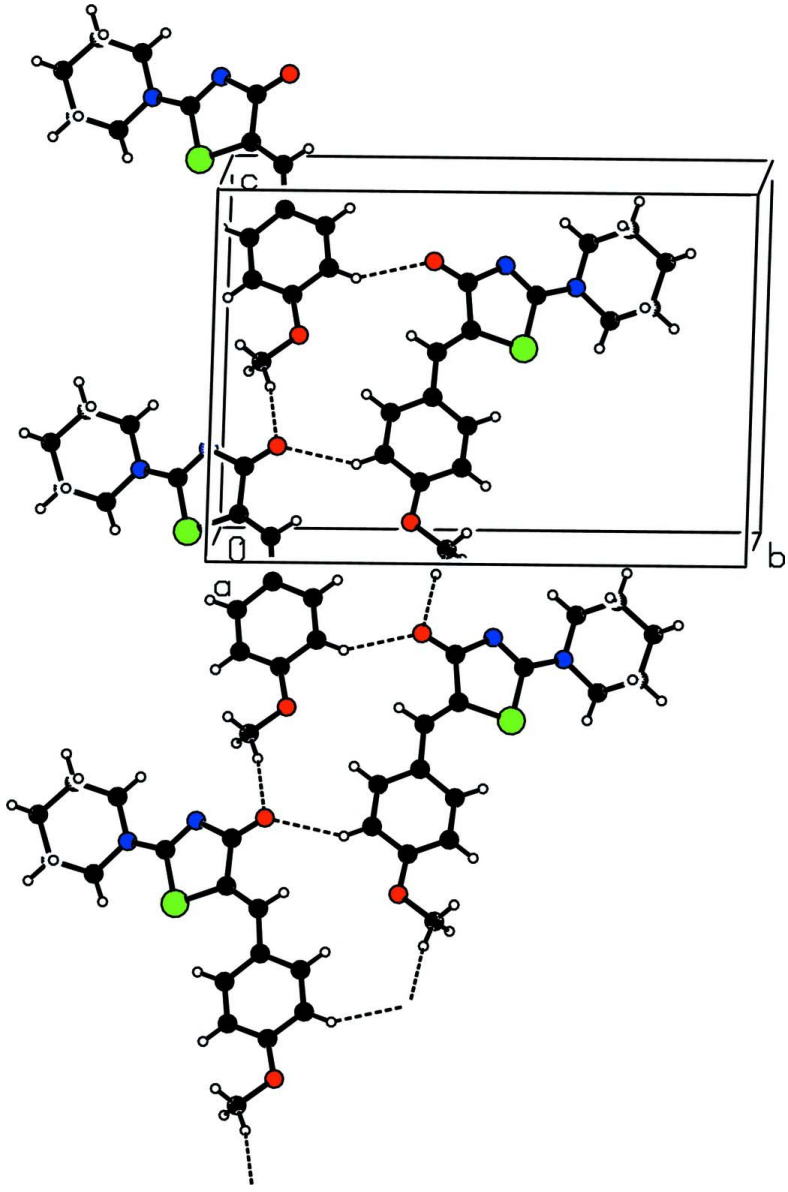

Figure 2

The crystal packing of the title compound, showing the molecules linked along the *c* axis. Hydrogen bonds (dashed lines) are shown.

(5*E*)-5-(4-Methoxybenzylidene)-2-(piperidin-1-yl)-1,3-thiazol- 4(5*H*)-one

Crystal data

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c = 12.4930 (4) Å
β = 121.518 (2)°
V = 1509.42 (9) Å^3
Z = 4
F(000) = 640
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6389 reflections
θ = 2.8–32.3°
μ = 0.22 mm^{-1}

T = 297 K
Block, brown

0.61 × 0.26 × 0.23 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.877, T_{max} = 0.950

20021 measured reflections
5432 independent reflections
4148 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 32.6°, θ_{min} = 2.3°
h = −12→12
k = −25→25
l = −17→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.02
5432 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.2092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.20003 (4)	0.555618 (16)	0.50684 (2)	0.03982 (9)
O1	0.39228 (18)	0.38959 (6)	0.75694 (9)	0.0638 (3)
O2	0.27412 (16)	0.35919 (6)	0.04461 (9)	0.0561 (2)
N1	0.28424 (15)	0.51913 (6)	0.73753 (9)	0.0425 (2)
N2	0.16266 (16)	0.64775 (6)	0.66938 (10)	0.0455 (2)
C1	0.32029 (16)	0.31624 (6)	0.34274 (10)	0.0382 (2)
H1A	0.3436	0.2711	0.3935	0.046*
C2	0.30857 (17)	0.30703 (7)	0.22911 (11)	0.0418 (2)
H2A	0.3216	0.2560	0.2034	0.050*
C3	0.27723 (16)	0.37401 (7)	0.15287 (11)	0.0409 (2)
C4	0.2523 (2)	0.44940 (7)	0.19023 (12)	0.0479 (3)
H4A	0.2292	0.4944	0.1392	0.057*
C5	0.2618 (2)	0.45754 (6)	0.30353 (12)	0.0457 (3)
H5A	0.2434	0.5084	0.3271	0.055*

C6	0.29786 (15)	0.39221 (6)	0.38362 (10)	0.0349 (2)
C7	0.31827 (16)	0.39806 (6)	0.50618 (10)	0.0374 (2)
H7A	0.3601	0.3509	0.5536	0.045*
C8	0.28758 (16)	0.45928 (6)	0.56330 (10)	0.0366 (2)
C9	0.32720 (18)	0.45128 (7)	0.69525 (11)	0.0422 (2)
C10	0.21678 (16)	0.57634 (6)	0.65225 (10)	0.0375 (2)
C11	0.1834 (2)	0.66894 (8)	0.79030 (12)	0.0500 (3)
H11A	0.2454	0.6255	0.8501	0.060*
H11B	0.0640	0.6764	0.7798	0.060*
C12	0.2935 (2)	0.74644 (8)	0.83907 (13)	0.0562 (3)
H12A	0.2980	0.7631	0.9151	0.067*
H12B	0.4178	0.7364	0.8601	0.067*
C13	0.2120 (2)	0.81400 (9)	0.74341 (16)	0.0675 (4)
H13A	0.2908	0.8611	0.7753	0.081*
H13B	0.0938	0.8289	0.7301	0.081*
C14	0.1894 (2)	0.78837 (8)	0.61928 (15)	0.0576 (3)
H14A	0.1271	0.8307	0.5574	0.069*
H14B	0.3088	0.7804	0.6300	0.069*
C15	0.08081 (19)	0.71084 (7)	0.57317 (12)	0.0489 (3)
H15A	−0.0437	0.7208	0.5517	0.059*
H15B	0.0770	0.6927	0.4980	0.059*
C16	0.2685 (3)	0.42727 (11)	−0.02815 (15)	0.0677 (4)
H16A	0.2886	0.4093	−0.0931	0.102*
H16B	0.3621	0.4652	0.0252	0.102*
H16C	0.1511	0.4530	−0.0654	0.102*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.05518 (18)	0.03126 (13)	0.03620 (14)	0.00014 (10)	0.02609 (12)	−0.00056 (9)
O1	0.1086 (9)	0.0433 (5)	0.0527 (5)	0.0192 (5)	0.0513 (6)	0.0128 (4)
O2	0.0807 (7)	0.0558 (5)	0.0460 (5)	0.0013 (5)	0.0430 (5)	−0.0033 (4)
N1	0.0585 (6)	0.0364 (4)	0.0369 (4)	−0.0010 (4)	0.0280 (4)	−0.0027 (3)
N2	0.0620 (6)	0.0336 (4)	0.0418 (5)	0.0007 (4)	0.0276 (5)	−0.0061 (4)
C1	0.0459 (6)	0.0306 (4)	0.0402 (5)	0.0027 (4)	0.0241 (5)	0.0004 (4)
C2	0.0517 (6)	0.0348 (5)	0.0436 (5)	0.0021 (4)	0.0282 (5)	−0.0047 (4)
C3	0.0470 (6)	0.0423 (5)	0.0392 (5)	−0.0020 (4)	0.0267 (5)	−0.0038 (4)
C4	0.0711 (8)	0.0361 (5)	0.0474 (6)	0.0014 (5)	0.0386 (6)	0.0040 (4)
C5	0.0724 (8)	0.0282 (4)	0.0497 (6)	0.0004 (5)	0.0411 (6)	−0.0009 (4)
C6	0.0410 (5)	0.0307 (4)	0.0364 (5)	−0.0024 (4)	0.0226 (4)	−0.0028 (3)
C7	0.0474 (6)	0.0308 (4)	0.0376 (5)	−0.0014 (4)	0.0248 (4)	−0.0004 (4)
C8	0.0461 (5)	0.0312 (4)	0.0357 (5)	−0.0037 (4)	0.0235 (4)	−0.0020 (4)
C9	0.0594 (7)	0.0352 (5)	0.0380 (5)	0.0000 (4)	0.0297 (5)	0.0006 (4)
C10	0.0447 (5)	0.0320 (4)	0.0364 (5)	−0.0058 (4)	0.0217 (4)	−0.0065 (4)
C11	0.0590 (7)	0.0486 (6)	0.0479 (6)	−0.0020 (5)	0.0319 (6)	−0.0128 (5)
C12	0.0565 (7)	0.0517 (7)	0.0551 (7)	−0.0024 (6)	0.0254 (6)	−0.0216 (6)
C13	0.0740 (10)	0.0401 (6)	0.0770 (10)	0.0010 (6)	0.0316 (8)	−0.0188 (6)
C14	0.0613 (8)	0.0362 (5)	0.0704 (9)	−0.0002 (5)	0.0311 (7)	−0.0004 (6)

C15	0.0561 (7)	0.0361 (5)	0.0490 (6)	0.0042 (5)	0.0236 (6)	−0.0020 (5)
C16	0.0943 (12)	0.0728 (10)	0.0530 (8)	−0.0026 (9)	0.0503 (8)	0.0041 (7)

Geometric parameters (Å, º)

S1—C8	1.7435 (11)	C7—C8	1.3407 (15)
S1—C10	1.7790 (11)	C7—H7A	0.9300
O1—C9	1.2225 (14)	C8—C9	1.5047 (15)
O2—C3	1.3609 (14)	C11—C12	1.5166 (19)
O2—C16	1.4309 (19)	C11—H11A	0.9700
N1—C10	1.3108 (14)	C11—H11B	0.9700
N1—C9	1.3691 (15)	C12—C13	1.513 (2)
N2—C10	1.3252 (14)	C12—H12A	0.9700
N2—C15	1.4634 (16)	C12—H12B	0.9700
N2—C11	1.4680 (16)	C13—C14	1.519 (2)
C1—C2	1.3781 (16)	C13—H13A	0.9700
C1—C6	1.4053 (14)	C13—H13B	0.9700
C1—H1A	0.9300	C14—C15	1.5096 (18)
C2—C3	1.3912 (16)	C14—H14A	0.9700
C2—H2A	0.9300	C14—H14B	0.9700
C3—C4	1.3852 (16)	C15—H15A	0.9700
C4—C5	1.3811 (17)	C15—H15B	0.9700
C4—H4A	0.9300	C16—H16A	0.9600
C5—C6	1.3919 (15)	C16—H16B	0.9600
C5—H5A	0.9300	C16—H16C	0.9600
C6—C7	1.4506 (15)		
C8—S1—C10	88.56 (5)	N2—C11—C12	109.25 (12)
C3—O2—C16	117.82 (11)	N2—C11—H11A	109.8
C10—N1—C9	111.72 (9)	C12—C11—H11A	109.8
C10—N2—C15	124.01 (10)	N2—C11—H11B	109.8
C10—N2—C11	120.94 (10)	C12—C11—H11B	109.8
C15—N2—C11	115.05 (10)	H11A—C11—H11B	108.3
C2—C1—C6	121.52 (10)	C13—C12—C11	111.81 (12)
C2—C1—H1A	119.2	C13—C12—H12A	109.3
C6—C1—H1A	119.2	C11—C12—H12A	109.3
C1—C2—C3	120.09 (10)	C13—C12—H12B	109.3
C1—C2—H2A	120.0	C11—C12—H12B	109.3
C3—C2—H2A	120.0	H12A—C12—H12B	107.9
O2—C3—C4	124.79 (11)	C12—C13—C14	111.12 (11)
O2—C3—C2	115.70 (10)	C12—C13—H13A	109.4
C4—C3—C2	119.52 (10)	C14—C13—H13A	109.4
C5—C4—C3	119.75 (11)	C12—C13—H13B	109.4
C5—C4—H4A	120.1	C14—C13—H13B	109.4
C3—C4—H4A	120.1	H13A—C13—H13B	108.0
C4—C5—C6	122.21 (10)	C15—C14—C13	110.46 (13)
C4—C5—H5A	118.9	C15—C14—H14A	109.6
C6—C5—H5A	118.9	C13—C14—H14A	109.6

C5—C6—C1	116.88 (10)	C15—C14—H14B	109.6
C5—C6—C7	124.46 (9)	C13—C14—H14B	109.6
C1—C6—C7	118.64 (9)	H14A—C14—H14B	108.1
C8—C7—C6	131.54 (10)	N2—C15—C14	110.80 (11)
C8—C7—H7A	114.2	N2—C15—H15A	109.5
C6—C7—H7A	114.2	C14—C15—H15A	109.5
C7—C8—C9	121.47 (10)	N2—C15—H15B	109.5
C7—C8—S1	129.49 (9)	C14—C15—H15B	109.5
C9—C8—S1	109.03 (7)	H15A—C15—H15B	108.1
O1—C9—N1	124.43 (11)	O2—C16—H16A	109.5
O1—C9—C8	122.07 (10)	O2—C16—H16B	109.5
N1—C9—C8	113.49 (9)	H16A—C16—H16B	109.5
N1—C10—N2	123.60 (10)	O2—C16—H16C	109.5
N1—C10—S1	117.18 (8)	H16A—C16—H16C	109.5
N2—C10—S1	119.22 (9)	H16B—C16—H16C	109.5

C6—C1—C2—C3	1.20 (18)	C7—C8—C9—O1	1.5 (2)
C16—O2—C3—C4	8.6 (2)	S1—C8—C9—O1	−178.96 (12)
C16—O2—C3—C2	−171.27 (13)	C7—C8—C9—N1	−178.78 (11)
C1—C2—C3—O2	177.84 (11)	S1—C8—C9—N1	0.74 (13)
C1—C2—C3—C4	−2.01 (19)	C9—N1—C10—N2	179.29 (12)
O2—C3—C4—C5	−178.76 (13)	C9—N1—C10—S1	−1.17 (14)
C2—C3—C4—C5	1.1 (2)	C15—N2—C10—N1	−177.59 (12)
C3—C4—C5—C6	0.7 (2)	C11—N2—C10—N1	2.90 (19)
C4—C5—C6—C1	−1.5 (2)	C15—N2—C10—S1	2.88 (17)
C4—C5—C6—C7	176.81 (12)	C11—N2—C10—S1	−176.63 (9)
C2—C1—C6—C5	0.53 (17)	C8—S1—C10—N1	1.37 (10)
C2—C1—C6—C7	−177.87 (11)	C8—S1—C10—N2	−179.07 (10)
C5—C6—C7—C8	9.2 (2)	C10—N2—C11—C12	123.69 (13)
C1—C6—C7—C8	−172.51 (12)	C15—N2—C11—C12	−55.87 (15)
C6—C7—C8—C9	−177.40 (11)	N2—C11—C12—C13	53.89 (16)
C6—C7—C8—S1	3.2 (2)	C11—C12—C13—C14	−54.80 (18)
C10—S1—C8—C7	178.38 (12)	C12—C13—C14—C15	54.15 (17)
C10—S1—C8—C9	−1.08 (9)	C10—N2—C15—C14	−122.75 (14)
C10—N1—C9—O1	179.94 (14)	C11—N2—C15—C14	56.79 (16)
C10—N1—C9—C8	0.25 (15)	C13—C14—C15—N2	−54.03 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···O1^{i}	0.93	2.48	3.3048 (16)	147
C5—H5*A*···S1	0.93	2.58	3.2809 (15)	132
C16—H16*A*···O1^{ii}	0.96	2.48	3.421 (3)	167

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