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(3*S*,4*Z*)-3-Chloro-1-methyl-4-[(2*E*)-(3-methylbenzylidene)hydrazinylidene]-3,4-dihydro-1*H*-2,1-benzothiazine 2,2-dioxide

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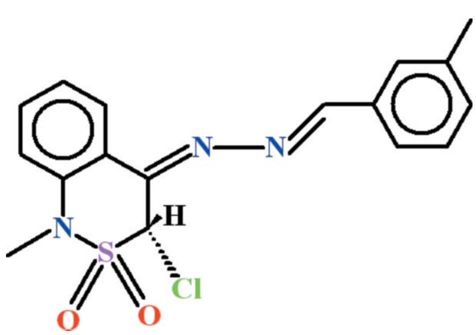
Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.004 Å; *R* factor = 0.036; *wR* factor = 0.088; data-to-parameter ratio = 19.5.

In the title compound, $C_{17}H_{16}ClN_3O_2S$, the dihedral angle between the benzene rings is 7.75 (13)°. The thiazine ring adopts an envelope conformation with the S atom as the flap at a distance of 0.813 (2) Å from the plane through the other five atoms. In the crystal, C—H···O hydrogen bonds link the molecules into chains propagating in [100].

Related literature

For related structures, see: Shafiq *et al.* (2011*a*,*b*,*c*). For further synthetic details, see: Shafiq *et al.* (2011*d*). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{17}H_{16}ClN_3O_2S$
M_r = 361.84
Orthorhombic, $P2_12_12_1$
a = 8.7734 (2) Å
b = 11.1271 (2) Å
c = 17.9423 (3) Å
V = 1751.57 (6) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.35 mm^{-1}
T = 296 K
0.26 × 0.18 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.930, T_{max} = 0.960
17152 measured reflections
4265 independent reflections
3478 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.088
S = 1.03
4265 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 1788 Friedel pairs
Flack parameter: 0.47 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.57	3.469 (3)	163
C10—H10···O1^{ii}	0.93	2.53	3.300 (3)	140

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6582).

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supporting information

Acta Cryst. (2012). E**68**, o338   [doi:10.1107/S1600536811056315]

(3*S*,4*Z*)-3-Chloro-1-methyl-4-[(2*E*)-(3-methylbenzylidene)hydrazinylidene]-3,4-dihydro-1*H*-2,1-benzothiazine 2,2-dioxide

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S1. Comment

The title compound (I), (Fig. 1) has been synthesized in continuation of our studies of Schiff bases (Shafiq *et al.*, 2011*a,b,c*).

In (I), the benzene rings A (C1—C6) and B (C11—C16) are planar with r. m. s. deviation of 0.0033 and 0.0002 Å, respectively. The dihedral angle between A/B is 7.75 (13)°. The central group C (N2/N3/C10) is of course planar. The dihedral angle between A/C and B/C is 6.02 (19) and 5.11 (21)°, respectively. The thiazine ring D (C1/C6/C9/C8/S1/N1) is in the nvelope form, with the maximum puckering amplitude (Cremer & Pople, 1975), Q = 0.5707 (16) Å. The molecules form one-dimensional polymeric chains extending along the *a*-axis due to H-bonding of C—H···O type (Table 1).

S2. Experimental

Schiff base derivative of (4*Z*)-4-hydrazinylidene-1-methyl-3,4-dihydro -1*H*-2,1-benzothiazine 2,2-dioxide and 3-methyl-benzaldehyde was prepared using the method reported previously (Shafiq *et al.* 2011*d*). The chlorination of the schiff base was undertaken using *N*-chloro succinimide and dibenzoylperoxide (Shafiq *et al.*, 2011*a*). The crude product was re-crystallized from ethyl acetate to yield orange needles of (I).

S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and x = 1.2 for aryl H-atoms.

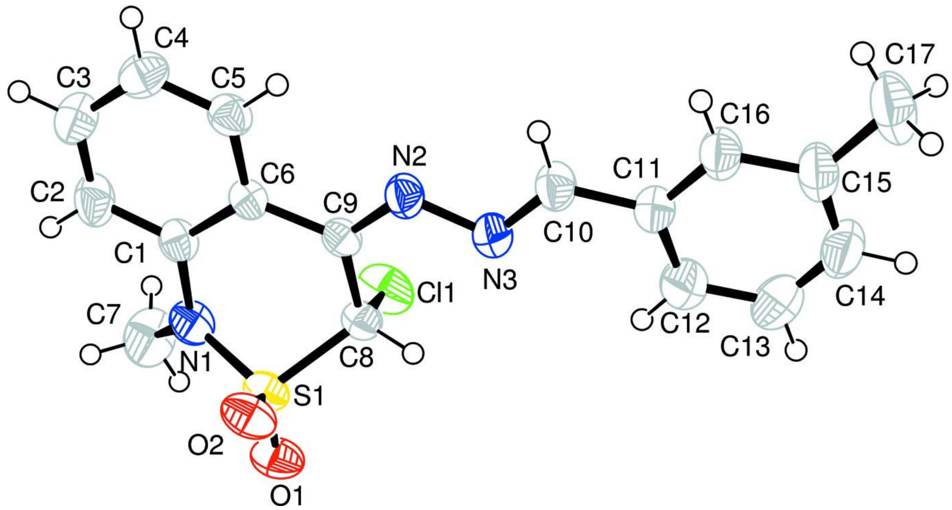

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level.

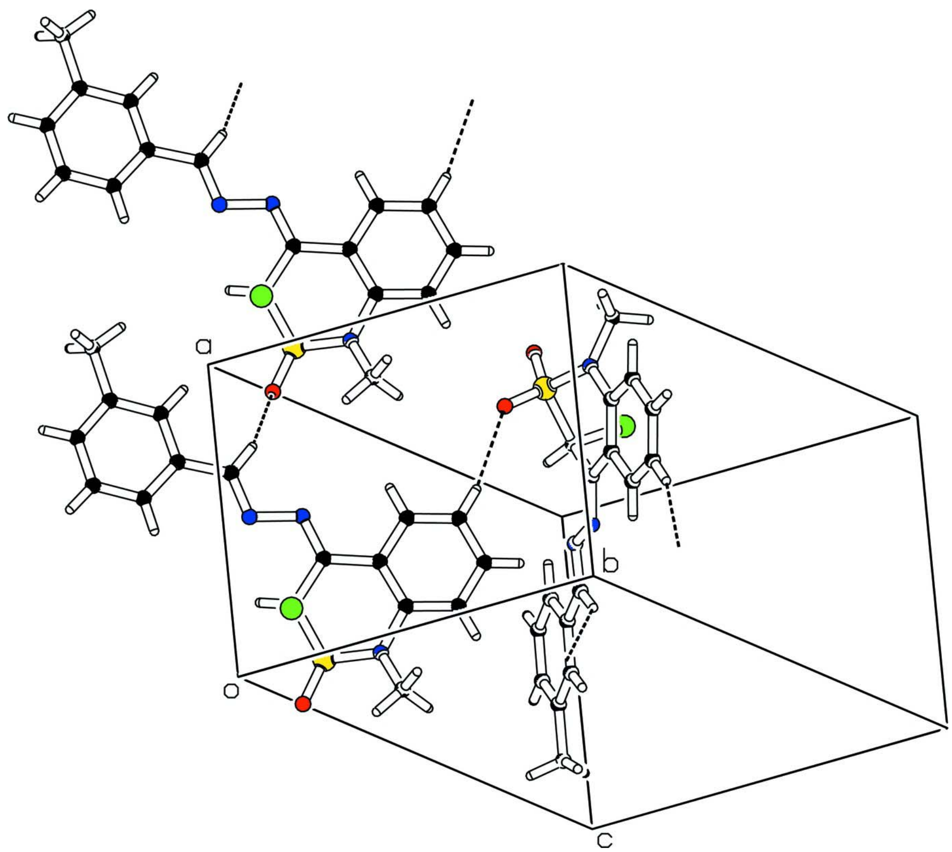

Figure 2

The partial packing showing chains extending along the [100] direction.

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a = 8.7734 (2) Å
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c = 17.9423 (3) Å
V = 1751.57 (6) Å^3
Z = 4
F(000) = 752
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2106 reflections
θ = 1.4–25.3°
μ = 0.35 mm^{-1}
T = 296 K
Needle, orange
0.26 × 0.18 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.930, T_{max} = 0.960
17152 measured reflections
4265 independent reflections
3478 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.3°, θ_{min} = 2.2°
h = −10→11
k = −14→14
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.088
S = 1.03
4265 reflections
219 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
$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.2645P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), **1788 Friedel pairs**
Absolute structure parameter: 0.47 (6)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
Cl1	0.16886 (7)	0.16151 (6)	0.00310 (3)	0.0619 (2)
S1	0.10071 (6)	0.09643 (5)	0.15565 (3)	0.0411 (2)

O1	−0.04053 (18)	0.05118 (15)	0.12847 (9)	0.0559 (6)
O2	0.17040 (19)	0.04041 (14)	0.21814 (8)	0.0524 (5)
N1	0.0827 (2)	0.23974 (17)	0.16940 (11)	0.0453 (6)
N2	0.5175 (2)	0.13115 (15)	0.09335 (10)	0.0410 (5)
N3	0.5229 (2)	0.03374 (16)	0.04310 (10)	0.0467 (6)
C1	0.2182 (3)	0.30519 (17)	0.18673 (11)	0.0381 (6)
C2	0.2065 (3)	0.4082 (2)	0.22991 (13)	0.0523 (8)
C3	0.3334 (3)	0.4739 (2)	0.24869 (13)	0.0531 (8)
C4	0.4751 (3)	0.4373 (2)	0.22482 (12)	0.0499 (8)
C5	0.4885 (3)	0.3360 (2)	0.18144 (11)	0.0422 (7)
C6	0.3614 (2)	0.26764 (17)	0.16138 (10)	0.0338 (6)
C7	−0.0609 (3)	0.3020 (3)	0.15644 (19)	0.0690 (10)
C8	0.2429 (2)	0.09333 (19)	0.08520 (10)	0.0380 (6)
C9	0.3815 (2)	0.16115 (17)	0.11258 (10)	0.0339 (6)
C10	0.6581 (3)	0.01494 (19)	0.02078 (12)	0.0455 (7)
C11	0.6974 (3)	−0.07862 (19)	−0.03287 (12)	0.0464 (7)
C12	0.5872 (3)	−0.1500 (3)	−0.06694 (14)	0.0668 (10)
C13	0.6332 (4)	−0.2383 (3)	−0.11655 (18)	0.0808 (13)
C14	0.7839 (4)	−0.2554 (2)	−0.13190 (16)	0.0751 (12)
C15	0.8968 (3)	−0.1863 (2)	−0.09916 (14)	0.0588 (8)
C16	0.8497 (3)	−0.0975 (2)	−0.04930 (12)	0.0513 (8)
C17	1.0632 (4)	−0.2042 (3)	−0.11637 (18)	0.0803 (11)
H2	0.11120	0.43326	0.24642	0.0628*
H3	0.32356	0.54297	0.27748	0.0637*
H4	0.56141	0.48092	0.23799	0.0598*
H5	0.58447	0.31247	0.16504	0.0506*
H7A	−0.10558	0.32386	0.20337	0.1034*
H7B	−0.12931	0.25007	0.12985	0.1034*
H7C	−0.04256	0.37318	0.12755	0.1034*
H8	0.27104	0.00984	0.07453	0.0456*
H10	0.73577	0.06310	0.03951	0.0545*
H12	0.48435	−0.13858	−0.05656	0.0802*
H13	0.56053	−0.28641	−0.13963	0.0969*
H14	0.81172	−0.31540	−0.16535	0.0899*
H16	0.92277	−0.04956	−0.02640	0.0616*
H17A	1.09454	−0.28229	−0.09956	0.1203*
H17B	1.12213	−0.14373	−0.09134	0.1203*
H17C	1.07907	−0.19801	−0.16917	0.1203*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0545 (4)	0.0921 (4)	0.0390 (3)	−0.0107 (3)	−0.0091 (3)	0.0114 (3)
S1	0.0304 (3)	0.0494 (3)	0.0434 (3)	−0.0036 (2)	0.0022 (2)	0.0065 (2)
O1	0.0360 (10)	0.0662 (10)	0.0655 (10)	−0.0139 (8)	−0.0005 (8)	0.0038 (8)
O2	0.0459 (10)	0.0616 (9)	0.0497 (8)	−0.0011 (8)	0.0023 (8)	0.0190 (7)
N1	0.0253 (10)	0.0544 (10)	0.0561 (11)	0.0053 (8)	0.0001 (8)	−0.0009 (8)
N2	0.0357 (10)	0.0437 (9)	0.0435 (9)	0.0038 (8)	0.0036 (8)	−0.0065 (7)

N3	0.0414 (12)	0.0459 (10)	0.0527 (10)	−0.0009 (9)	0.0088 (9)	−0.0098 (8)
C1	0.0322 (11)	0.0447 (11)	0.0374 (10)	0.0049 (9)	−0.0006 (8)	−0.0003 (8)
C2	0.0417 (13)	0.0592 (13)	0.0561 (13)	0.0133 (12)	0.0043 (11)	−0.0130 (12)
C3	0.0569 (17)	0.0487 (12)	0.0537 (12)	0.0045 (12)	−0.0028 (12)	−0.0115 (10)
C4	0.0488 (15)	0.0507 (12)	0.0501 (12)	−0.0065 (11)	−0.0107 (11)	−0.0038 (10)
C5	0.0331 (12)	0.0520 (12)	0.0414 (10)	−0.0031 (10)	0.0002 (9)	−0.0009 (9)
C6	0.0301 (11)	0.0399 (9)	0.0315 (9)	0.0022 (8)	−0.0007 (8)	0.0017 (8)
C7	0.0372 (15)	0.0779 (19)	0.092 (2)	0.0183 (12)	−0.0136 (14)	−0.0157 (17)
C8	0.0340 (11)	0.0412 (10)	0.0389 (10)	−0.0015 (9)	0.0017 (9)	0.0008 (9)
C9	0.0285 (11)	0.0386 (9)	0.0347 (9)	0.0022 (9)	0.0005 (8)	0.0035 (8)
C10	0.0416 (13)	0.0473 (12)	0.0475 (11)	0.0076 (10)	0.0017 (10)	−0.0071 (9)
C11	0.0489 (14)	0.0422 (11)	0.0481 (11)	0.0029 (11)	0.0066 (10)	−0.0052 (9)
C12	0.0661 (18)	0.0677 (16)	0.0667 (16)	−0.0120 (15)	0.0121 (14)	−0.0195 (14)
C13	0.093 (3)	0.0663 (17)	0.083 (2)	−0.0196 (17)	0.0097 (18)	−0.0307 (15)
C14	0.110 (3)	0.0500 (14)	0.0652 (16)	0.0004 (17)	0.0260 (17)	−0.0173 (12)
C15	0.0755 (18)	0.0493 (13)	0.0516 (12)	0.0193 (14)	0.0202 (14)	0.0017 (10)
C16	0.0553 (15)	0.0465 (12)	0.0521 (12)	0.0091 (12)	0.0076 (11)	−0.0046 (10)
C17	0.084 (2)	0.080 (2)	0.0770 (19)	0.0358 (17)	0.0277 (17)	0.0024 (16)

Geometric parameters (Å, º)

Cl1—C8	1.7797 (19)	C12—C13	1.386 (4)
S1—O1	1.4237 (17)	C13—C14	1.364 (5)
S1—O2	1.4211 (16)	C14—C15	1.385 (4)
S1—N1	1.621 (2)	C15—C16	1.396 (3)
S1—C8	1.7763 (19)	C15—C17	1.505 (4)
N1—C1	1.428 (3)	C2—H2	0.9300
N1—C7	1.457 (3)	C3—H3	0.9300
N2—N3	1.411 (2)	C4—H4	0.9300
N2—C9	1.286 (2)	C5—H5	0.9300
N3—C10	1.269 (3)	C7—H7A	0.9600
C1—C2	1.387 (3)	C7—H7B	0.9600
C1—C6	1.400 (3)	C7—H7C	0.9600
C2—C3	1.374 (4)	C8—H8	0.9800
C3—C4	1.377 (4)	C10—H10	0.9300
C4—C5	1.375 (3)	C12—H12	0.9300
C5—C6	1.397 (3)	C13—H13	0.9300
C6—C9	1.484 (3)	C14—H14	0.9300
C8—C9	1.513 (3)	C16—H16	0.9300
C10—C11	1.459 (3)	C17—H17A	0.9600
C11—C12	1.393 (4)	C17—H17B	0.9600
C11—C16	1.384 (4)	C17—H17C	0.9600
O1—S1—O2	119.32 (10)	C16—C15—C17	120.8 (2)
O1—S1—N1	108.37 (10)	C11—C16—C15	122.0 (2)
O1—S1—C8	111.14 (9)	C1—C2—H2	119.00
O2—S1—N1	110.69 (10)	C3—C2—H2	119.00
O2—S1—C8	104.52 (9)	C2—C3—H3	120.00

N1—S1—C8	101.29 (10)	C4—C3—H3	120.00
S1—N1—C1	116.97 (14)	C3—C4—H4	120.00
S1—N1—C7	121.86 (17)	C5—C4—H4	120.00
C1—N1—C7	120.8 (2)	C4—C5—H5	119.00
N3—N2—C9	113.71 (16)	C6—C5—H5	119.00
N2—N3—C10	111.08 (17)	N1—C7—H7A	109.00
N1—C1—C2	118.8 (2)	N1—C7—H7B	110.00
N1—C1—C6	121.60 (17)	N1—C7—H7C	109.00
C2—C1—C6	119.6 (2)	H7A—C7—H7B	109.00
C1—C2—C3	121.1 (2)	H7A—C7—H7C	109.00
C2—C3—C4	119.9 (2)	H7B—C7—H7C	109.00
C3—C4—C5	119.7 (2)	Cl1—C8—H8	110.00
C4—C5—C6	121.6 (2)	S1—C8—H8	110.00
C1—C6—C5	118.04 (18)	C9—C8—H8	109.00
C1—C6—C9	122.46 (17)	N3—C10—H10	118.00
C5—C6—C9	119.47 (17)	C11—C10—H10	118.00
Cl1—C8—S1	108.93 (10)	C11—C12—H12	121.00
Cl1—C8—C9	110.45 (14)	C13—C12—H12	121.00
S1—C8—C9	108.89 (13)	C12—C13—H13	120.00
N2—C9—C6	118.41 (16)	C14—C13—H13	120.00
N2—C9—C8	121.94 (17)	C13—C14—H14	119.00
C6—C9—C8	119.63 (15)	C15—C14—H14	119.00
N3—C10—C11	123.1 (2)	C11—C16—H16	119.00
C10—C11—C12	122.2 (2)	C15—C16—H16	119.00
C10—C11—C16	118.5 (2)	C15—C17—H17A	109.00
C12—C11—C16	119.4 (2)	C15—C17—H17B	109.00
C11—C12—C13	119.0 (3)	C15—C17—H17C	109.00
C12—C13—C14	120.7 (3)	H17A—C17—H17B	109.00
C13—C14—C15	122.0 (3)	H17A—C17—H17C	109.00
C14—C15—C16	117.0 (2)	H17B—C17—H17C	109.00
C14—C15—C17	122.2 (2)		

O1—S1—N1—C1	−171.41 (15)	C1—C2—C3—C4	−0.3 (3)
O1—S1—N1—C7	1.7 (2)	C2—C3—C4—C5	0.9 (3)
O2—S1—N1—C1	55.99 (18)	C3—C4—C5—C6	−0.8 (3)
O2—S1—N1—C7	−130.9 (2)	C4—C5—C6—C1	0.1 (3)
C8—S1—N1—C1	−54.42 (17)	C4—C5—C6—C9	178.17 (19)
C8—S1—N1—C7	118.7 (2)	C1—C6—C9—N2	−178.30 (18)
O1—S1—C8—Cl1	49.97 (14)	C1—C6—C9—C8	3.4 (3)
O1—S1—C8—C9	170.49 (13)	C5—C6—C9—N2	3.7 (3)
O2—S1—C8—Cl1	179.94 (12)	C5—C6—C9—C8	−174.64 (17)
O2—S1—C8—C9	−59.54 (15)	Cl1—C8—C9—N2	−93.1 (2)
N1—S1—C8—Cl1	−64.97 (12)	Cl1—C8—C9—C6	85.20 (18)
N1—S1—C8—C9	55.54 (15)	S1—C8—C9—N2	147.38 (16)
S1—N1—C1—C2	−151.50 (17)	S1—C8—C9—C6	−34.4 (2)
S1—N1—C1—C6	28.4 (3)	N3—C10—C11—C12	3.8 (4)
C7—N1—C1—C2	35.3 (3)	N3—C10—C11—C16	−175.3 (2)
C7—N1—C1—C6	−144.8 (2)	C10—C11—C12—C13	−179.1 (2)

C9—N2—N3—C10	174.07 (18)	C16—C11—C12—C13	0.0 (4)
N3—N2—C9—C6	−176.04 (16)	C10—C11—C16—C15	179.1 (2)
N3—N2—C9—C8	2.2 (3)	C12—C11—C16—C15	0.0 (3)
N2—N3—C10—C11	−178.60 (19)	C11—C12—C13—C14	0.0 (4)
N1—C1—C2—C3	179.5 (2)	C12—C13—C14—C15	0.0 (5)
C6—C1—C2—C3	−0.4 (3)	C13—C14—C15—C16	0.0 (4)
N1—C1—C6—C5	−179.34 (18)	C13—C14—C15—C17	−179.5 (3)
N1—C1—C6—C9	2.6 (3)	C14—C15—C16—C11	0.0 (3)
C2—C1—C6—C5	0.5 (3)	C17—C15—C16—C11	179.6 (2)
C2—C1—C6—C9	−177.55 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.57	3.469 (3)	163
C10—H10···O1^{ii}	0.93	2.53	3.300 (3)	140

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