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(*Z*)-3-(2-Chlorobenzyl)-1,5-benzothiazepin-4(5*H*)-one

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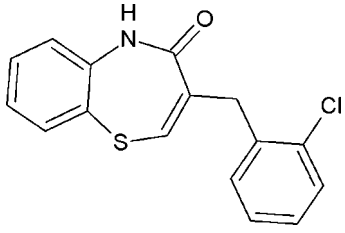
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.129; data-to-parameter ratio = 19.4.

In the crystal structure of the title compound, $C_{16}H_{12}ClNOS$, the molecules are linked into centrosymmetric $R_2^2(8)$ dimers *via* pairs of N—H···O hydrogen bonds. The seven-membered ring adopts a boat conformation.

Related literature

For the pharmaceutical properties of thiazepin derivatives, see: Tomascovic *et al.* (2000); Rajsner *et al.* (1971); Metys *et al.* (1965). For conformations of thiazepin derivatives, see: Huang *et al.* (2011). For graph-set analysis of hydrogen bonds, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{12}ClNOS$
M_r = 301.78
Triclinic, $P\overline{1}$
a = 8.4958 (3) Å
b = 8.7197 (3) Å
c = 10.0520 (3) Å
α = 101.930 (1)°
β = 95.179 (2)°
γ = 90.314 (2)°
V = 725.38 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 K
0.32 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.980, T_{max} = 0.990
10314 measured reflections
3596 independent reflections
2961 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 0.93
3596 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.82 (2)	2.08 (2)	2.8911 (19)	174 (3)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5944).

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supplementary materials

Acta Cryst. (2012). E**68**, o2283 [doi:10.1107/S1600536812029030]

(*Z*)-3-(2-Chlorobenzyl)-1,5-benzothiazepin-4(5*H*)-one

V. Sabari, R. Selvakumar, M. Bakthadoss and S. Aravindhan

Comment

The title compound is used as an intermediate for the synthesis of dosulepin, which is an antidepressant of the tricyclic family. Dosulepin prevents reabsorbing of serotonin and noradrenaline in the brain, helps to prolong the mood lightening effect of any released noradrenaline and serotonin, thus relieving depression. The dibenzo[c,e]thiazepin derivatives exhibit chiroptical properties (Tomascovic *et al.*, 2000). Dibenzo[b,e]thiazepin-5,5-dioxide derivatives possess antihistaminic and antiallergenic activities (Rajsner *et al.*, 1971). Benzene thiazepin derivatives are identified as a new type of effective antihistaminic compounds (Metys *et al.*, 1965). Considering the wide range of biological activities of the thiazepin derivatives,we determined the crystal structure of the title compound.

X-Ray analysis confirms the molecular structure and atom connectivity as illustrated in (Fig. 1). The seven membered thiazepin ring adopts a boat conformation (Huang *et al.*, 2011). The sum of the bond angles around the N1 atom (357.72°) indicates sp^2 hybridization. The molecules are linked *via* N—H···O hydrogen bonds to centrosymmetric dimers with graph set notation $R_2^2(8)$ (Bernstein *et al.*, 1995) (Fig.2).

Experimental

A mixture of (*Z*)-methyl 2-(bromomethyl)-3-(2-chlorophenyl)acrylate (2 mmol) and *o*-aminothiophenol (2 mmol) in the presence of potassium *tert*-butoxide (4.8 mmol) in dry THF (10 ml) was stirred at room temperature for 1 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 ml) and extracted with ethyl acetate (3 *x* 20 ml). The organic layer was washed with brine (2 *x* 20 ml) and dried over anhydrous sodium sulfate. The organic layer was concentrated, which successfully provide the crude final product ((*Z*)-3-(2-chlorobenzyl)benzo[*b*][1,4]thiazepin-4(5*H*)-one). The final product was purified by column chromatography on silica gel to afford the title compound in 45% yields.

Refinement

H atoms bonded to C were refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C)] using a riding model with C—H ranging from 0.93 Å to 0.97 Å. The amino H atom was freely refined.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

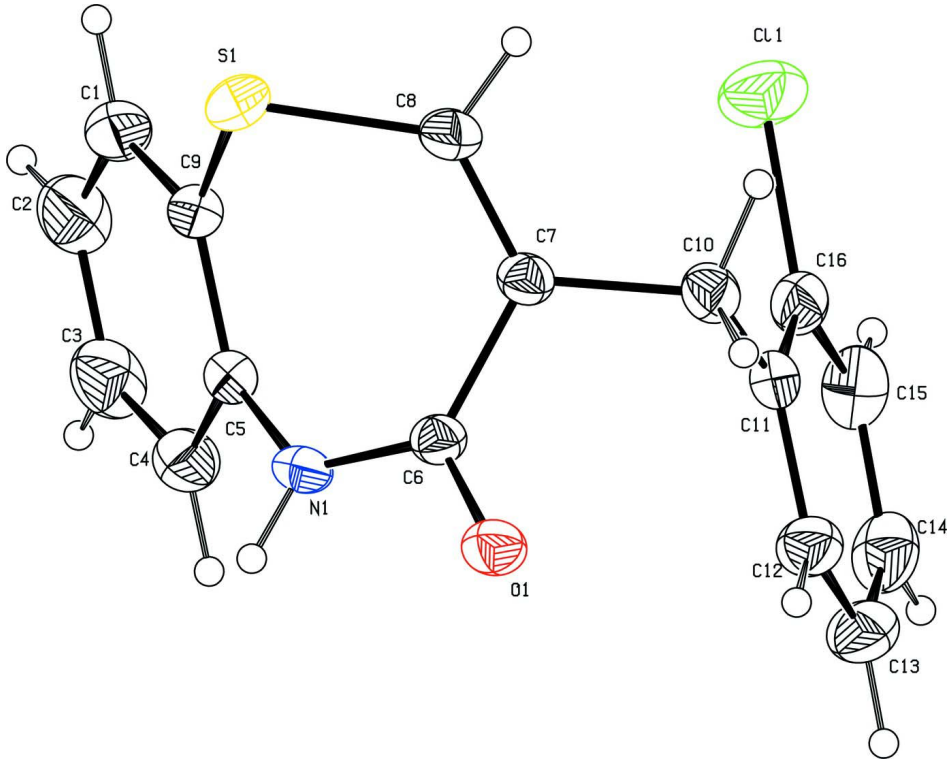

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

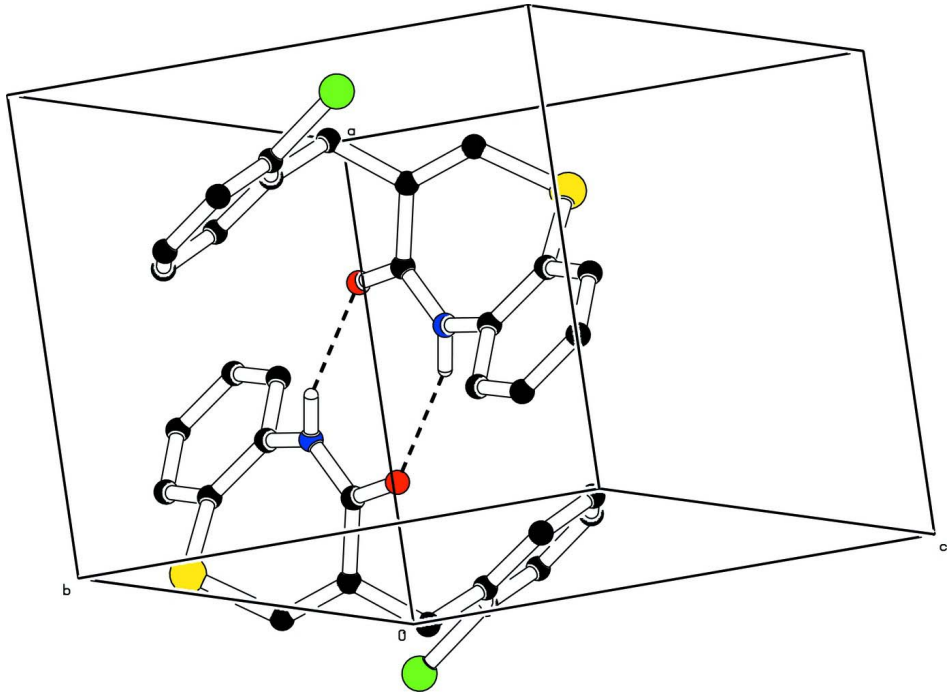

Figure 2

A view of the crystal packing. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

(*Z*)-3-(2-Chlorobenzyl)-1,5-benzothiazepin-4(5*H*)-one

Crystal data

$C_{16}H_{12}ClNOS$
$M_r = 301.78$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.4958$ (3) Å
$b = 8.7197$ (3) Å
$c = 10.0520$ (3) Å
$\alpha = 101.930$ (1)°
$\beta = 95.179$ (2)°
$\gamma = 90.314$ (2)°
$V = 725.38$ (4) Å^3
$Z = 2$
$F(000) = 312$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8725 reflections
$\theta = 2.8$–$29.1°$
$\mu = 0.40$ mm^{-1}
$T = 298$ K
Triclinic, colourless
0.32 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 15.9948 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.980$, $T_{max} = 0.990$
10314 measured reflections
3596 independent reflections
2961 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 28.6°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 10$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.129$
$S = 0.93$
3596 reflections
185 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
$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.3987P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ 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}
C1	0.3986 (3)	0.7811 (3)	0.4385 (2)	0.0611 (6)
H1	0.3329	0.7850	0.3602	0.073*

C2	0.5362 (3)	0.6996 (3)	0.4275 (3)	0.0727 (7)
H2	0.5640	0.6500	0.3421	0.087*
C3	0.6327 (3)	0.6915 (3)	0.5429 (3)	0.0686 (6)
H3	0.7259	0.6362	0.5354	0.082*
C4	0.5921 (2)	0.7650 (3)	0.6696 (2)	0.0554 (5)
H4	0.6578	0.7587	0.7474	0.066*
C5	0.45365 (19)	0.8482 (2)	0.68197 (17)	0.0400 (4)
N1	0.42278 (17)	0.92803 (19)	0.81405 (14)	0.0425 (3)
C6	0.29024 (18)	0.92977 (19)	0.87848 (16)	0.0352 (3)
C7	0.13755 (18)	0.85995 (19)	0.80480 (17)	0.0361 (3)
C8	0.0826 (2)	0.8806 (2)	0.68177 (19)	0.0455 (4)
H8	−0.0194	0.8430	0.6497	0.055*
C9	0.3563 (2)	0.8580 (2)	0.56553 (17)	0.0429 (4)
C10	0.0416 (2)	0.7741 (2)	0.88754 (19)	0.0435 (4)
H10A	0.0236	0.8440	0.9730	0.052*
H10B	−0.0604	0.7426	0.8376	0.052*
C11	0.12551 (19)	0.6308 (2)	0.91707 (17)	0.0382 (4)
C12	0.2153 (2)	0.6381 (2)	1.0410 (2)	0.0506 (4)
H12	0.2198	0.7312	1.1062	0.061*
C13	0.2978 (3)	0.5119 (3)	1.0702 (2)	0.0615 (6)
H13	0.3578	0.5212	1.1538	0.074*
C14	0.2922 (3)	0.3731 (3)	0.9772 (3)	0.0622 (6)
H14	0.3486	0.2882	0.9970	0.075*
C15	0.2030 (3)	0.3594 (3)	0.8545 (2)	0.0594 (5)
H15	0.1969	0.2646	0.7914	0.071*
C16	0.1220 (2)	0.4874 (2)	0.82485 (18)	0.0466 (4)
Cl1	0.01021 (9)	0.46470 (9)	0.66795 (6)	0.0789 (2)
O1	0.29428 (15)	0.98862 (16)	1.00167 (12)	0.0457 (3)
S1	0.18502 (6)	0.97237 (7)	0.57440 (5)	0.05265 (16)
H1A	0.501 (3)	0.958 (3)	0.867 (2)	0.051 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0598 (12)	0.0839 (16)	0.0365 (9)	−0.0198 (11)	−0.0013 (8)	0.0081 (9)
C2	0.0641 (14)	0.0927 (18)	0.0539 (13)	−0.0154 (13)	0.0192 (11)	−0.0075 (12)
C3	0.0482 (11)	0.0836 (17)	0.0714 (15)	0.0037 (11)	0.0188 (10)	0.0043 (12)
C4	0.0397 (9)	0.0745 (14)	0.0536 (11)	0.0014 (9)	0.0038 (8)	0.0170 (10)
C5	0.0358 (8)	0.0482 (10)	0.0363 (8)	−0.0078 (7)	0.0001 (6)	0.0113 (7)
N1	0.0328 (7)	0.0581 (9)	0.0340 (7)	−0.0079 (6)	−0.0066 (6)	0.0076 (6)
C6	0.0359 (7)	0.0334 (8)	0.0362 (8)	−0.0005 (6)	−0.0041 (6)	0.0105 (6)
C7	0.0308 (7)	0.0366 (8)	0.0408 (8)	0.0011 (6)	−0.0018 (6)	0.0104 (6)
C8	0.0348 (8)	0.0536 (11)	0.0492 (10)	−0.0023 (7)	−0.0088 (7)	0.0186 (8)
C9	0.0420 (8)	0.0497 (10)	0.0373 (8)	−0.0107 (7)	−0.0029 (7)	0.0130 (7)
C10	0.0333 (8)	0.0494 (10)	0.0496 (10)	0.0012 (7)	0.0043 (7)	0.0139 (8)
C11	0.0347 (7)	0.0421 (9)	0.0396 (8)	−0.0047 (6)	0.0059 (6)	0.0118 (7)
C12	0.0593 (11)	0.0457 (10)	0.0452 (10)	−0.0068 (8)	−0.0056 (8)	0.0103 (8)
C13	0.0661 (13)	0.0602 (13)	0.0611 (13)	−0.0029 (10)	−0.0127 (10)	0.0279 (11)
C14	0.0671 (13)	0.0521 (12)	0.0759 (15)	0.0095 (10)	0.0129 (11)	0.0299 (11)
C15	0.0749 (14)	0.0442 (11)	0.0595 (12)	0.0017 (10)	0.0226 (11)	0.0051 (9)

C16	0.0504 (10)	0.0523 (11)	0.0373 (8)	−0.0065 (8)	0.0080 (7)	0.0082 (7)
Cl1	0.0987 (5)	0.0856 (5)	0.0433 (3)	−0.0106 (4)	−0.0118 (3)	0.0007 (3)
O1	0.0450 (7)	0.0514 (7)	0.0370 (6)	−0.0043 (5)	−0.0015 (5)	0.0029 (5)
S1	0.0507 (3)	0.0646 (3)	0.0485 (3)	0.0007 (2)	−0.0081 (2)	0.0308 (2)

Geometric parameters (Å, º)

C1—C2	1.372 (4)	C8—S1	1.7554 (19)
C1—C9	1.393 (3)	C8—H8	0.9300
C1—H1	0.9300	C9—S1	1.767 (2)
C2—C3	1.373 (4)	C10—C11	1.510 (3)
C2—H2	0.9300	C10—H10A	0.9700
C3—C4	1.378 (3)	C10—H10B	0.9700
C3—H3	0.9300	C11—C12	1.390 (3)
C4—C5	1.387 (3)	C11—C16	1.393 (3)
C4—H4	0.9300	C12—C13	1.376 (3)
C5—C9	1.389 (2)	C12—H12	0.9300
C5—N1	1.414 (2)	C13—C14	1.366 (3)
N1—C6	1.347 (2)	C13—H13	0.9300
N1—H1A	0.81 (2)	C14—C15	1.371 (3)
C6—O1	1.236 (2)	C14—H14	0.9300
C6—C7	1.494 (2)	C15—C16	1.384 (3)
C7—C8	1.330 (2)	C15—H15	0.9300
C7—C10	1.513 (2)	C16—Cl1	1.740 (2)
C2—C1—C9	120.8 (2)	C5—C9—S1	121.50 (14)
C2—C1—H1	119.6	C1—C9—S1	119.38 (15)
C9—C1—H1	119.6	C11—C10—C7	111.24 (14)
C3—C2—C1	119.9 (2)	C11—C10—H10A	109.4
C3—C2—H2	120.0	C7—C10—H10A	109.4
C1—C2—H2	120.0	C11—C10—H10B	109.4
C2—C3—C4	120.2 (2)	C7—C10—H10B	109.4
C2—C3—H3	119.9	H10A—C10—H10B	108.0
C4—C3—H3	119.9	C12—C11—C16	116.03 (17)
C3—C4—C5	120.4 (2)	C12—C11—C10	120.21 (16)
C3—C4—H4	119.8	C16—C11—C10	123.74 (16)
C5—C4—H4	119.8	C13—C12—C11	122.1 (2)
C4—C5—C9	119.62 (17)	C13—C12—H12	118.9
C4—C5—N1	117.68 (16)	C11—C12—H12	118.9
C9—C5—N1	122.60 (17)	C14—C13—C12	120.3 (2)
C6—N1—C5	129.94 (14)	C14—C13—H13	119.8
C6—N1—H1A	112.3 (15)	C12—C13—H13	119.8
C5—N1—H1A	115.5 (16)	C13—C14—C15	119.7 (2)
O1—C6—N1	119.72 (14)	C13—C14—H14	120.2
O1—C6—C7	118.76 (15)	C15—C14—H14	120.2
N1—C6—C7	121.52 (14)	C14—C15—C16	119.7 (2)
C8—C7—C6	123.87 (16)	C14—C15—H15	120.1
C8—C7—C10	121.87 (15)	C16—C15—H15	120.1
C6—C7—C10	114.06 (14)	C15—C16—C11	122.12 (18)
C7—C8—S1	125.94 (14)	C15—C16—Cl1	118.14 (16)

C7—C8—H8	117.0	C11—C16—Cl1	119.73 (15)
S1—C8—H8	117.0	C8—S1—C9	99.34 (8)
C5—C9—C1	119.06 (19)		

C9—C1—C2—C3	0.9 (4)	C2—C1—C9—S1	175.81 (18)
C1—C2—C3—C4	−0.1 (4)	C8—C7—C10—C11	119.48 (19)
C2—C3—C4—C5	−0.2 (4)	C6—C7—C10—C11	−65.55 (19)
C3—C4—C5—C9	−0.4 (3)	C7—C10—C11—C12	96.9 (2)
C3—C4—C5—N1	−176.6 (2)	C7—C10—C11—C16	−81.8 (2)
C4—C5—N1—C6	−131.9 (2)	C16—C11—C12—C13	0.9 (3)
C9—C5—N1—C6	52.0 (3)	C10—C11—C12—C13	−177.81 (19)
C5—N1—C6—O1	169.22 (17)	C11—C12—C13—C14	−0.7 (3)
C5—N1—C6—C7	−9.9 (3)	C12—C13—C14—C15	−0.4 (4)
O1—C6—C7—C8	136.80 (19)	C13—C14—C15—C16	1.3 (3)
N1—C6—C7—C8	−44.0 (3)	C14—C15—C16—C11	−1.1 (3)
O1—C6—C7—C10	−38.0 (2)	C14—C15—C16—Cl1	−179.98 (17)
N1—C6—C7—C10	141.13 (17)	C12—C11—C16—C15	0.0 (3)
C6—C7—C8—S1	7.7 (3)	C10—C11—C16—C15	178.67 (17)
C10—C7—C8—S1	−177.85 (14)	C12—C11—C16—Cl1	178.87 (14)
C4—C5—C9—C1	1.2 (3)	C10—C11—C16—Cl1	−2.4 (2)
N1—C5—C9—C1	177.26 (17)	C7—C8—S1—C9	55.28 (19)
C4—C5—C9—S1	−176.03 (15)	C5—C9—S1—C8	−59.33 (16)
N1—C5—C9—S1	0.0 (2)	C1—C9—S1—C8	123.43 (16)
C2—C1—C9—C5	−1.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.82 (2)	2.08 (2)	2.8911 (19)	174 (3)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+2.