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(*Z*)-2-(5-Chloro-2-oxoindolin-3-ylidene)-*N*-methylhydrazinecarbothioamide

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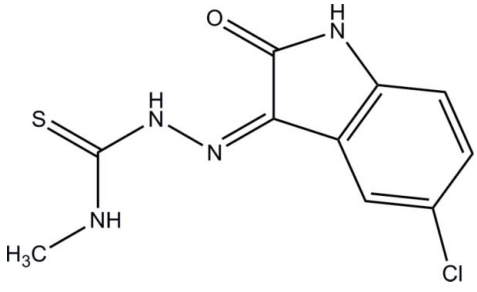
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.078; data-to-parameter ratio = 29.3.

In the title compound, $C_{10}H_9ClN_4OS$, an intramolecular N—H···O hydrogen-bonding interaction and an N—H···N interaction generate ring motifs [graph sets $S(6)$ and $S(5)$, respectively]. In the crystal, molecules form a chain through N—H···O hydrogen bonds, and these are extended by N—H···S hydrogen-bonding interactions into an infinite three-dimensional network. The crystal structure also exhibits weak C—H···π interactions.

Related literature

For related structures, see: Qasem Ali *et al.* (2012, 2011*a*,*b*); Ali *et al.* (2012). For various biological activities of Schiff bases, see: Bhandari *et al.* (2008); Bhardwaj *et al.* (2010); Pandeya *et al.* (1999); Sridhar *et al.* (2002); Suryavanshi & Pai (2006). For cytotoxic and anticancer activities of isatin and its derivatives, see: Vine *et al.* (2009). For graph-set analysis, see Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_9ClN_4OS$
M_r = 268.72
Orthorhombic, $P2_12_12_1$
a = 6.2558 (1) Å
b = 10.1449 (1) Å
c = 18.5682 (2) Å
V = 1178.42 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 100 K
0.34 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.853, T_{max} = 0.961
16807 measured reflections
4886 independent reflections
4072 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.078
S = 1.05
4886 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 2074 Friedel pairs
Flack parameter: 0.01 (5)

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

*Cg*2 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1N4···N2	0.88 (2)	2.27 (2)	2.6416 (18)	105.6 (15)
N4—H1N4···S1^{i}	0.88 (2)	2.70 (2)	3.4972 (13)	152.2 (16)
N3—H1N3···O1	0.86 (2)	2.086 (19)	2.7526 (16)	134.3 (17)
N1—H1N1···O1^{ii}	0.81 (2)	2.01 (2)	2.8161 (16)	175 (2)
C3—H3*A*···*Cg*2^{iii}	0.95	2.59	3.38	141

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+2$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: ZS2177).

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supplementary materials

Acta Cryst. (2012). E**68**, o964–o965 [doi:10.1107/S1600536812007386]

(*Z*)-2-(5-Chloro-2-oxoindolin-3-ylidene)-*N*-methylhydrazinecarbothioamide

Amna Qasem Ali, Naser Eltaher Eltayeb, Siang Guan Teoh, Abdussalam Salhin and Hoong-Kun Fun

Comment

Isatin (2,3-dioxindole) is an endogenous compound identified in humans, and its effect has been studied in a variety of systems. Biological properties of isatin and its derivatives include a range of actions in the brain, offer protection against bacterial (Suryavanshi & Pai, 2006) and fungal infections and possess anticonvulsant, anti-HIV (Pandeya *et al.*, 1999), anti-depressant and anti-inflammatory activities (Bhandari *et al.*, 2008). Recently, we reported the crystal structure of (*Z*)-2-(5-chloro-2-oxoindolin-3-ylidene)-*N*-methylhydrazinecarbothioamide (Qasem Ali *et al.*, 2012). In the present paper we describe the single-crystal X-ray diffraction study of title compound, $C_{10}H_9ClN_4OS$ (Fig. 1).

In this compound, the chain N2/N3/C9/S1/N4/C10 is connected to the nine-membered 5-chloroindolin-2-one ring system at C7. In this chain C7—N2—N3—C9 and C10—N4—C9—S1 torsion angles are -177.77 (13)° and 2.7 (2)°, respectively. The essentially planar conformation of the molecule is maintained by the cyclic intramolecular N3—H1N3···O1 hydrogen-bonding interaction together with the N4—H1N4···N2 interaction [graph sets *S*(6) and *S*(5), respectively (Bernstein *et al.*, 1995)] (Table 1).

In the crystal the molecules form chain substructures through intermolecular N1—H1N1···O1 hydrogen bonds and these are extended by N4—H1N4···S1 hydrogen-bonding interactions into an infinite a three-dimensional network (Table 1, Fig. 2). Weak C—H···π interactions are also present [C3—H···*Cg*2^{iii} = 3.38 Å], where *Cg*2 is the centroid of the C1—C6 ring. For symmetry code (iii), see Table 1.

Experimental

The Schiff base has been synthesized by refluxing the reaction mixture of a hot ethanolic solution (30 ml) of 5-methyl-3-thiosemicarbazide (0.01 mol) and a hot ethanolic solution (30 ml) of 5-chloroisatin (0.01 mol) for 2 hr. The precipitate formed during reflux was filtered, washed with cold ethanol and recrystallized from hot ethanol. Yield (m.p.): 85% (568.4–569.0 K). The yellow crystals were grown in ethylacetate–DMF (3:1) by slow evaporation at room temperature.

Refinement

Nitrogen bound H atoms were located in a difference Fourier map and were refined freely. The remaining H atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å (aromatic ring) and C—H = 0.98 Å (methyl group) with U_{iso}(H) = 1.2U_{eq}(aromatic C) and U_{iso}(H) = 1.5U_{eq}(methyl C). The highest residual electron density peak (0.41 eÅ^{-3}) is located at 0.73 Å from Cl1 and the deepest hole (-0.32 eÅ^{-3}) is located at 0.59 Å from Sl.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication:

SHELXTL (Sheldrick, 2008) and *PLATON* (Spek, 2009).

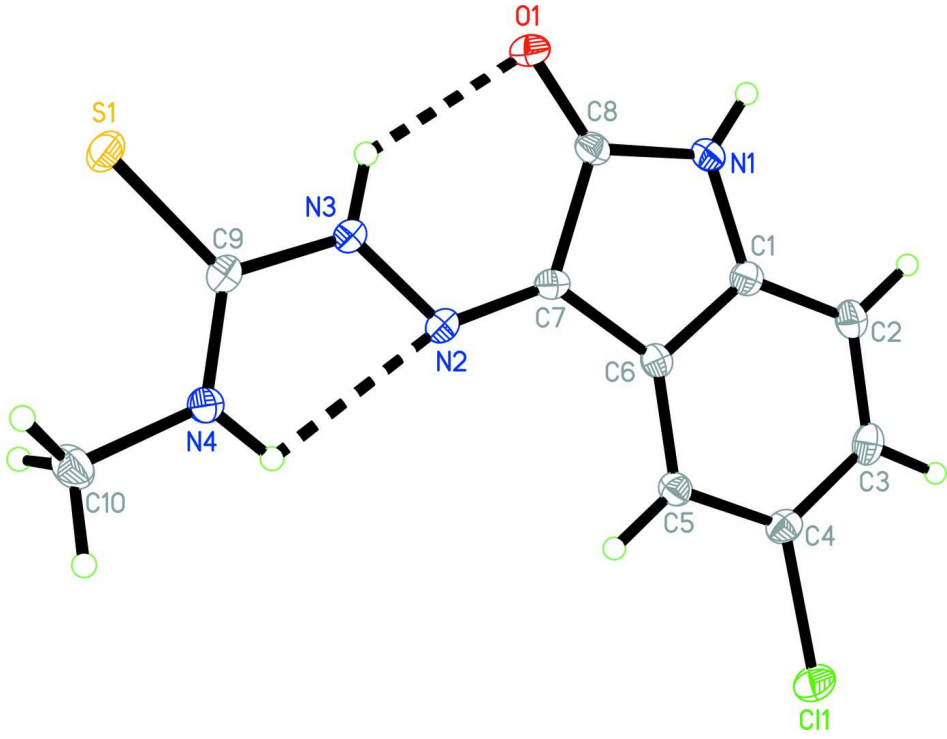

Figure 1

The molecular structure and the atom-numbering scheme of the title compound, with 50% probability displacement ellipsoids.

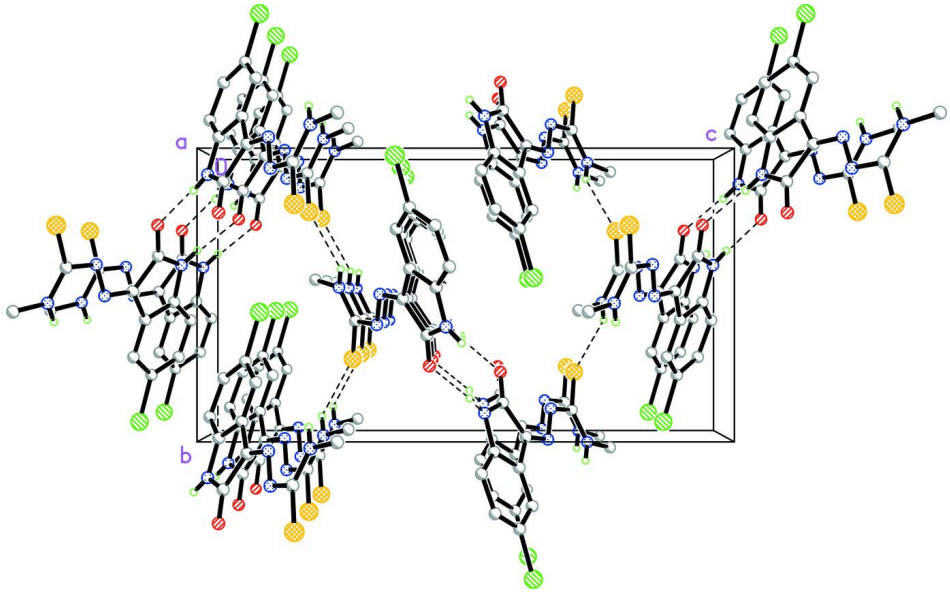

Figure 2

The crystal packing of the title compound viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

(*Z*)-2-(5-Chloro-2-oxoindolin-3-ylidene)-*N*- methylhydrazinecarbothioamide

Crystal data

$C_{10}H_9ClN_4OS$
$M_r = 268.72$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.2558$ (1) Å
$b = 10.1449$ (1) Å
$c = 18.5682$ (2) Å
$V = 1178.42$ (3) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.515$ Mg m^{-3}
Melting point = 568.4–569.0 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5715 reflections
$\theta = 3.0$–$34.1°$
$\mu = 0.49$ mm^{-1}
$T = 100$ K
Needle, yellow
0.34 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.853$, $T_{max} = 0.961$
16807 measured reflections
4886 independent reflections
4072 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 34.4°$, $\theta_{min} = 2.2°$
$h = -8 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.078$
$S = 1.05$
4886 reflections
167 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
$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 0.1624P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Absolute structure: Flack (1983), 2074 Friedel pairs
Flack parameter: 0.01 (5)

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}
Cl1	−0.30754 (7)	0.94253 (3)	0.877263 (19)	0.02071 (9)
S1	0.74171 (6)	0.27814 (3)	0.785567 (19)	0.01723 (8)
O1	0.15801 (18)	0.26544 (10)	0.93453 (5)	0.0176 (2)

N1	−0.1313 (2)	0.39745 (12)	0.96526 (6)	0.0154 (3)
N2	0.2697 (2)	0.50267 (11)	0.84352 (6)	0.0131 (2)
N3	0.4038 (2)	0.39983 (12)	0.83590 (6)	0.0144 (2)
N4	0.6135 (2)	0.52656 (12)	0.76196 (7)	0.0147 (2)
C1	−0.2019 (3)	0.52525 (13)	0.94808 (7)	0.0138 (3)
C2	−0.3841 (3)	0.58915 (15)	0.97154 (7)	0.0160 (3)
H2A	−0.4830	0.5474	1.0029	0.019*
C3	−0.4167 (2)	0.71814 (15)	0.94706 (7)	0.0163 (3)
H3A	−0.5411	0.7650	0.9613	0.020*
C4	−0.2680 (3)	0.77827 (13)	0.90194 (7)	0.0148 (3)
C5	−0.0871 (2)	0.71336 (14)	0.87730 (7)	0.0139 (3)
H5A	0.0114	0.7551	0.8458	0.017*
C6	−0.0562 (2)	0.58458 (14)	0.90075 (7)	0.0124 (3)
C7	0.1086 (2)	0.48782 (13)	0.88630 (7)	0.0127 (3)
C8	0.0532 (3)	0.36840 (14)	0.93058 (7)	0.0139 (3)
C9	0.5824 (2)	0.41081 (13)	0.79306 (7)	0.0127 (3)
C10	0.7903 (3)	0.55324 (15)	0.71346 (8)	0.0201 (3)
H10A	0.8041	0.6486	0.7065	0.030*
H10B	0.7634	0.5107	0.6670	0.030*
H10C	0.9228	0.5184	0.7342	0.030*
H1N4	0.516 (4)	0.588 (2)	0.7671 (10)	0.033 (6)*
H1N3	0.383 (3)	0.328 (2)	0.8590 (10)	0.032 (6)*
H1N1	−0.189 (4)	0.3462 (19)	0.9928 (11)	0.035 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0223 (2)	0.01646 (14)	0.02338 (15)	0.00725 (15)	−0.00058 (16)	0.00258 (13)
S1	0.01584 (18)	0.01392 (14)	0.02193 (15)	0.00320 (15)	0.00197 (16)	−0.00174 (13)
O1	0.0214 (6)	0.0140 (4)	0.0174 (4)	0.0042 (5)	−0.0002 (4)	0.0032 (4)
N1	0.0183 (7)	0.0138 (5)	0.0141 (5)	−0.0012 (5)	0.0033 (5)	0.0017 (4)
N2	0.0134 (6)	0.0118 (4)	0.0140 (5)	0.0016 (5)	−0.0007 (5)	−0.0013 (4)
N3	0.0150 (7)	0.0122 (5)	0.0161 (5)	0.0026 (5)	0.0026 (5)	0.0008 (4)
N4	0.0129 (6)	0.0138 (5)	0.0175 (5)	0.0005 (5)	0.0018 (5)	0.0017 (4)
C1	0.0155 (7)	0.0135 (5)	0.0123 (5)	−0.0014 (5)	−0.0011 (6)	−0.0011 (4)
C2	0.0145 (7)	0.0189 (6)	0.0148 (6)	−0.0014 (6)	0.0018 (6)	−0.0014 (5)
C3	0.0130 (7)	0.0201 (6)	0.0159 (6)	0.0021 (6)	−0.0002 (6)	−0.0040 (5)
C4	0.0157 (7)	0.0142 (5)	0.0146 (5)	0.0022 (6)	−0.0034 (6)	−0.0002 (5)
C5	0.0144 (7)	0.0146 (5)	0.0128 (5)	0.0000 (6)	0.0003 (6)	0.0013 (5)
C6	0.0119 (7)	0.0145 (6)	0.0108 (5)	−0.0006 (5)	−0.0009 (5)	−0.0003 (4)
C7	0.0142 (7)	0.0109 (5)	0.0129 (6)	0.0000 (5)	−0.0013 (5)	0.0010 (4)
C8	0.0168 (7)	0.0135 (6)	0.0113 (5)	−0.0019 (6)	−0.0007 (6)	0.0010 (4)
C9	0.0115 (7)	0.0129 (5)	0.0136 (6)	0.0000 (5)	−0.0023 (5)	−0.0026 (5)
C10	0.0162 (8)	0.0229 (7)	0.0211 (6)	−0.0025 (6)	0.0021 (6)	0.0042 (6)

Geometric parameters (Å, º)

Cl1—C4	1.7458 (14)	C1—C6	1.402 (2)
S1—C9	1.6806 (14)	C2—C3	1.400 (2)
O1—C8	1.2354 (18)	C2—H2A	0.9500

N1—C8	1.355 (2)	C3—C4	1.392 (2)
N1—C1	1.4062 (18)	C3—H3A	0.9500
N1—H1N1	0.81 (2)	C4—C5	1.387 (2)
N2—C7	1.292 (2)	C5—C6	1.3906 (19)
N2—N3	1.3463 (17)	C5—H5A	0.9500
N3—C9	1.376 (2)	C6—C7	1.449 (2)
N3—H1N3	0.86 (2)	C7—C8	1.5044 (19)
N4—C9	1.3229 (18)	C10—H10A	0.9800
N4—C10	1.4520 (19)	C10—H10B	0.9800
N4—H1N4	0.88 (2)	C10—H10C	0.9800
C1—C2	1.382 (2)		

C8—N1—C1	111.11 (12)	C4—C5—C6	117.14 (13)
C8—N1—H1N1	122.4 (16)	C4—C5—H5A	121.4
C1—N1—H1N1	126.4 (16)	C6—C5—H5A	121.4
C7—N2—N3	117.41 (11)	C5—C6—C1	120.62 (14)
N2—N3—C9	120.27 (12)	C5—C6—C7	132.66 (13)
N2—N3—H1N3	121.0 (14)	C1—C6—C7	106.73 (12)
C9—N3—H1N3	118.7 (14)	N2—C7—C6	126.15 (12)
C9—N4—C10	123.25 (13)	N2—C7—C8	127.55 (13)
C9—N4—H1N4	119.0 (14)	C6—C7—C8	106.30 (12)
C10—N4—H1N4	117.5 (14)	O1—C8—N1	127.44 (13)
C2—C1—C6	122.18 (13)	O1—C8—C7	126.25 (14)
C2—C1—N1	128.31 (14)	N1—C8—C7	106.31 (12)
C6—C1—N1	109.52 (13)	N4—C9—N3	116.33 (13)
C1—C2—C3	117.14 (14)	N4—C9—S1	125.98 (12)
C1—C2—H2A	121.4	N3—C9—S1	117.69 (10)
C3—C2—H2A	121.4	N4—C10—H10A	109.5
C4—C3—C2	120.50 (14)	N4—C10—H10B	109.5
C4—C3—H3A	119.7	H10A—C10—H10B	109.5
C2—C3—H3A	119.7	N4—C10—H10C	109.5
C5—C4—C3	122.37 (13)	H10A—C10—H10C	109.5
C5—C4—Cl1	118.82 (11)	H10B—C10—H10C	109.5
C3—C4—Cl1	118.79 (12)		

C7—N2—N3—C9	−177.77 (13)	N3—N2—C7—C6	−178.53 (13)
C8—N1—C1—C2	178.80 (14)	N3—N2—C7—C8	1.2 (2)
C8—N1—C1—C6	−0.92 (16)	C5—C6—C7—N2	−2.2 (3)
C6—C1—C2—C3	−1.2 (2)	C1—C6—C7—N2	177.97 (14)
N1—C1—C2—C3	179.15 (13)	C5—C6—C7—C8	177.99 (15)
C1—C2—C3—C4	−1.0 (2)	C1—C6—C7—C8	−1.83 (15)
C2—C3—C4—C5	2.3 (2)	C1—N1—C8—O1	179.09 (15)
C2—C3—C4—Cl1	−176.16 (11)	C1—N1—C8—C7	−0.27 (16)
C3—C4—C5—C6	−1.3 (2)	N2—C7—C8—O1	2.1 (3)
Cl1—C4—C5—C6	177.14 (11)	C6—C7—C8—O1	−178.07 (14)
C4—C5—C6—C1	−0.9 (2)	N2—C7—C8—N1	−178.50 (14)
C4—C5—C6—C7	179.33 (14)	C6—C7—C8—N1	1.30 (15)
C2—C1—C6—C5	2.1 (2)	C10—N4—C9—N3	−178.06 (13)
N1—C1—C6—C5	−178.12 (13)	C10—N4—C9—S1	2.7 (2)

C2—C1—C6—C7	−178.02 (13)	N2—N3—C9—N4	1.08 (19)
N1—C1—C6—C7	1.72 (15)	N2—N3—C9—S1	−179.58 (10)

Hydrogen-bond geometry (Å, º)

*Cg*2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1*N*4···N2	0.88 (2)	2.27 (2)	2.6416 (18)	105.6 (15)
N4—H1*N*4···S1^{i}	0.88 (2)	2.70 (2)	3.4972 (13)	152.2 (16)
N3—H1*N*3···O1	0.86 (2)	2.086 (19)	2.7526 (16)	134.3 (17)
N1—H1*N*1···O1^{ii}	0.81 (2)	2.01 (2)	2.8161 (16)	175 (2)
C3—H3*A*···*Cg*2^{iii}	0.95	2.59	3.38	141

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*−1/2, −*y*+1/2, −*z*+2; (iii) *x*−1/2, −*y*+3/2, −*z*+2.