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N′-[(*E*)-2-Hydroxy-5-iodobenzylidene]-4-methylbenzenesulfonohydrazide

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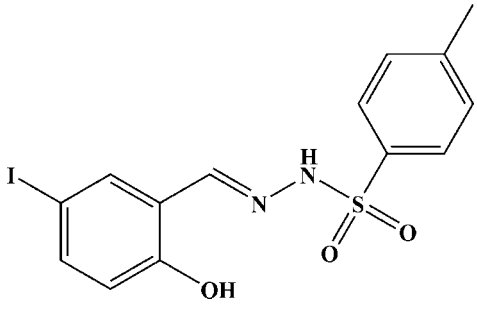
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.046; wR factor = 0.114; data-to-parameter ratio = 19.5.

In the title molecule, $C_{14}H_{13}IN_2O_3S$, the dihedral angle between the planes of the benzene and toluene rings is 84.3 (3)°. The molecule displays a *trans* conformation with respect to the C=N bond. There is an intramolecular O—H···N hydrogen bond with the azomethine N atom as acceptor. In the crystal, N—H···O hydrogen bonds connect the molecules into chains running along the *b* axis.

Related literature

For background to sulfonamides, see: Kayser *et al.* (2004). For related structures and their applications, see: Shahverdizadeh *et al.* (2011); Ali *et al.* (2007); Tierney *et al.* (2006); Silva *et al.* (2006). For polymorphism in sulfonohydrazides, see: Kia *et al.* (2008); Tai *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{13}IN_2O_3S$
M_r = 416.23
Monoclinic, $P2_1$
a = 6.2467 (12) Å
b = 10.394 (2) Å
c = 11.971 (2) Å
β = 92.42 (3)°
V = 776.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.21 mm^{-1}
T = 298 K
0.50 × 0.40 × 0.20 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.405, T_{max} = 0.667
6035 measured reflections
3841 independent reflections
2791 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.114
S = 0.92
3841 reflections
197 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 e Å^{-3}
Absolute structure: Flack (1983), 1641 Friedel pairs
Flack parameter: −0.06 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82 (2)	1.91 (5)	2.589 (6)	139 (7)
N2—H2A···O1^{i}	0.86 (2)	2.05 (2)	2.914 (6)	173 (6)

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5993).

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N′-[(*E*)-2-Hydroxy-5-iodobenzylidene]-4-methylbenzenesulfonohydrazide

Massomeh Ghorbanloo and Behrouz Notash

S1. Comment

Sulfonyl hydrazones are found to exhibit large medicinal applications. Similar to sulfonamides, sulfonyl hydrazones also have various biological activities (Kayser *et al.*, 2004). For example, imidosulfonylhydrazones have antibacterial and antineociceptive properties (Silva *et al.*, 2006). Acidic sulfonyl hydrazone derivatives have analgesic and anti-inflammatory activities. On the other hand, polymorphism is a phenomenon wherein the same substances exhibits different crystal packing arrangements and is of practical importance *e.g.*, pharmaceutical processes where different physical properties of polymorphic forms can substantially alter the viability and quality of product. Plymorphism is another interesting subject in sulfonyl hydrazones. sulfonyl hydrazones derived from the condensation of *O*-hydroxy aldehydes and sulfonyl acid hydrazides can form different polymorphs. Kia *et al.* (2008) and Tai *et al.* (2009) have reported two polymorph of these type of compounds.

We report here the crystal structure of (*E*)-*N*′-(2-hydroxy-5-iodobenzylidene)-4-methylbenzenesulfonohydrazide. The asymmetric unit of the title compound contains one molecule, which is shown in Fig. 1. Bond distances and bond angles are in the normal range of similar compounds (Shahverdizadeh *et al.*, 2011; Ali *et al.*, 2007; Tierney *et al.*, 2006). The molecule displays *trans* configuration with respect to the C=N bond. The packing diagram of the title compound is shown in Fig. 2. In the title compound, the dihedral angle between the planes of benzene and toluene rings is 84.3 (3)°. There is an intramolecular O—H···N hydrogen bond in which the nitrogen of the azomethine group (–C=N–) acting as hydrogen bond acceptor. Intermolecular N—H···O hydrogen bond stabilize the crystal structure (Fig. 2 & Table 1).

S2. Experimental

For preparing the title compound, a methanol (10 ml) solution of 2-hydroxy-5-iodobenzaldehyde (2 mmol) was dropwise added to a methanol solution (10 ml) of 4-methyl-benzenesulfonic acid hydrazide (2 mmol), and the mixture was refluxed for 3 hrs. Then the solution was evaporated on a steam bath to 5 ml and cooled to room temperature. A white precipitate of the title compound was separated and filtered off, washed with 5 ml of cooled methanol and then dried in air. X-ray quality crystals of the title compound were obtained from methanol by slow solvent evaporation. Yield: 90%. Selected IR (cm^{-1}): 3464 (w, broad), 3140 (*m*), 1619 (*s*), 1481 (*vs*), 1359 (*s*), 1329 (*vs*), 1264 (*vs*), 1177 (*s*), 1087 (*s*), 956 (*vs*), 869 (*vs*) 772 (*s*), 666 (*s*), 545 (*s*), 458 (*m*).

S3. Refinement

The hydrogen atoms bonded to O and N atoms were found in difference Fourier map and there coordinates were refined with *U*iso(H) = 1.2 *U*eq(O,N). The O—H and N—H distances were restrained to 0.82 (2)Å and 0.86 (2)Å, respectively. H atoms bonded to C were positioned geometrically and refined as riding atoms with C—H = 0.96 Å and *U*iso(H) = 1.5 *U*eq(C) for the methyl group and C—H = 0.93 Å and *U*iso(H) = 1.2 U_{eq}(C) for the other H atoms.

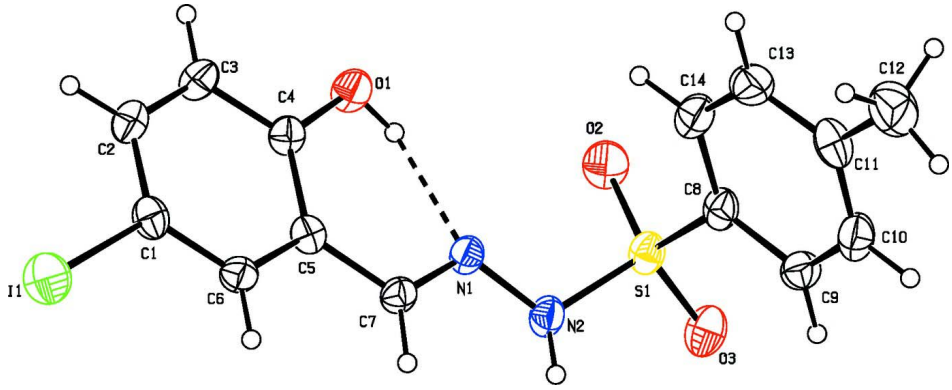

Figure 1

The molecular structure of the title compound with atom labels. Anisotropic displacement ellipsoids drawn at 30% probability level for non-H atoms.

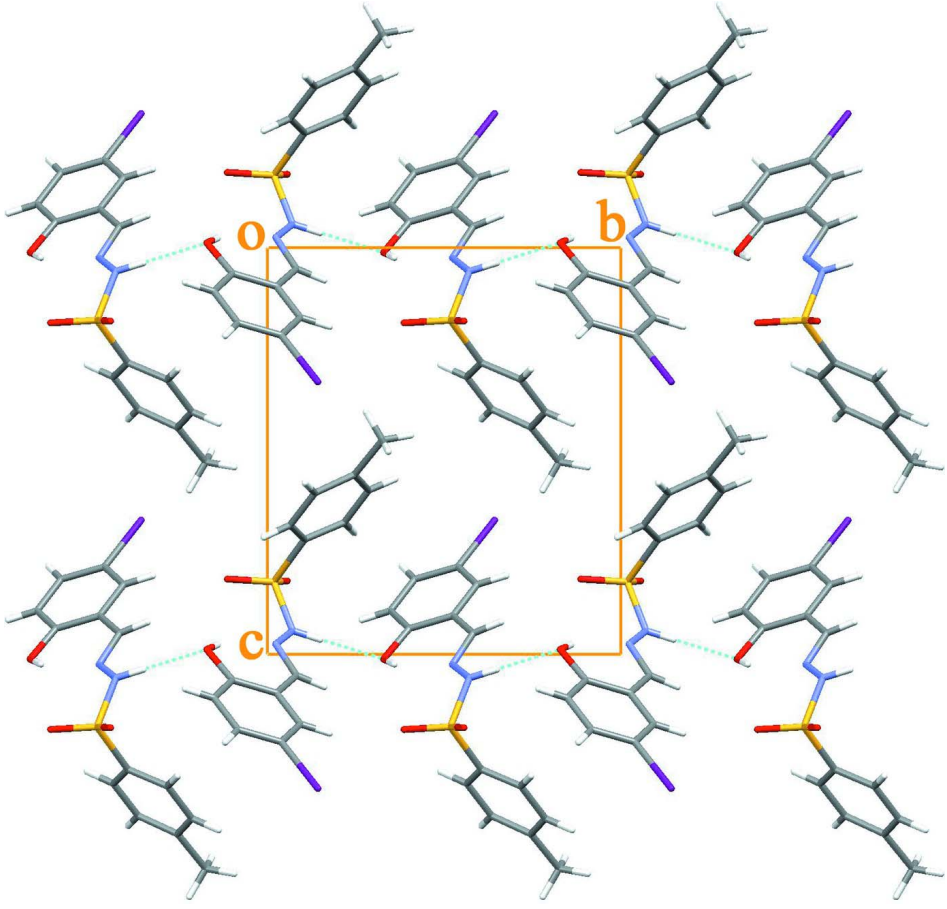

Figure 2

The packing diagram of the title compound. Hydrogen bonds are shown as blue dashed line.

N′-[(*E*)-2-Hydroxy-5-iodobenzylidene]-4- methylbenzenesulfonohydrazide

Crystal data

$C_{14}H_{13}IN_2O_3S$
$M_r = 416.23$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.2467$ (12) Å
$b = 10.394$ (2) Å
$c = 11.971$ (2) Å
$\beta = 92.42$ (3)°
$V = 776.6$ (3) Å^3
$Z = 2$
$F(000) = 408$
$D_x = 1.780$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3841 reflections
$\theta = 1.7$–29.2°
$\mu = 2.21$ mm^{-1}
$T = 298$ K
Plate, colorless
0.50 × 0.40 × 0.20 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 mm pixels mm^{-1}
rotation method scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
$T_{min} = 0.405$, $T_{max} = 0.667$
6035 measured reflections
3841 independent reflections
2791 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 29.2°$, $\theta_{min} = 1.7°$
$h = -7 \rightarrow 8$
$k = -14 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.114$
$S = 0.92$
3841 reflections
197 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.071P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.99$ e Å^{-3}
Absolute structure: Flack (1983), 1641 Friedel pairs
Absolute structure parameter: −0.06 (3)

Special details

Experimental. shape of crystal determined optically

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}
I1	0.80088 (6)	0.64270 (4)	−0.32973 (4)	0.07048 (17)

S1	−0.2369 (2)	0.51804 (13)	0.17826 (10)	0.0423 (3)
O1	0.2908 (7)	0.3287 (4)	0.0117 (4)	0.0493 (9)
O2	−0.1752 (7)	0.3869 (4)	0.1850 (4)	0.0584 (11)
O3	−0.4564 (6)	0.5561 (4)	0.1806 (4)	0.0566 (10)
N1	0.0354 (7)	0.5237 (4)	0.0251 (3)	0.0387 (9)
N2	−0.1570 (7)	0.5714 (4)	0.0580 (4)	0.0427 (10)
C1	0.6247 (8)	0.5391 (5)	−0.2153 (4)	0.0437 (12)
C2	0.6906 (8)	0.4168 (6)	−0.1848 (4)	0.0452 (12)
H2	0.8103	0.3806	−0.2161	0.054*
C3	0.5781 (9)	0.3485 (5)	−0.1074 (5)	0.0455 (12)
H3	0.6245	0.2672	−0.0849	0.055*
C4	0.3963 (8)	0.4012 (5)	−0.0633 (4)	0.0377 (10)
C5	0.3257 (8)	0.5256 (5)	−0.0950 (4)	0.0369 (10)
C6	0.4431 (8)	0.5934 (5)	−0.1716 (4)	0.0406 (11)
H6	0.3999	0.6755	−0.1937	0.049*
C7	0.1345 (8)	0.5822 (5)	−0.0518 (4)	0.0374 (10)
H7	0.0837	0.6605	−0.0795	0.045*
C8	−0.0891 (8)	0.6054 (5)	0.2828 (4)	0.0420 (12)
C9	−0.1739 (10)	0.7173 (5)	0.3242 (5)	0.0456 (12)
H9	−0.3088	0.7457	0.2992	0.055*
C10	−0.0552 (11)	0.7865 (6)	0.4035 (5)	0.0543 (15)
H10	−0.1116	0.8620	0.4317	0.065*
C11	0.1458 (11)	0.7461 (6)	0.4418 (5)	0.0543 (14)
C12	0.2764 (15)	0.8255 (10)	0.5268 (7)	0.082 (2)
H12A	0.2910	0.7791	0.5960	0.123*
H12B	0.2049	0.9058	0.5390	0.123*
H12C	0.4158	0.8419	0.4991	0.123*
C13	0.2264 (9)	0.6346 (9)	0.3970 (5)	0.0598 (14)
H13	0.3618	0.6065	0.4214	0.072*
C14	0.1134 (10)	0.5641 (6)	0.3177 (5)	0.0532 (14)
H14	0.1717	0.4899	0.2880	0.064*
H1	0.173 (6)	0.359 (7)	0.024 (6)	0.064*
H2A	−0.186 (11)	0.649 (3)	0.037 (6)	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0614 (2)	0.0783 (3)	0.0737 (3)	−0.0046 (3)	0.02653 (18)	0.0203 (3)
S1	0.0421 (7)	0.0388 (6)	0.0469 (6)	−0.0040 (5)	0.0134 (5)	−0.0008 (5)
O1	0.057 (2)	0.0328 (19)	0.060 (2)	0.0003 (17)	0.019 (2)	0.0067 (16)
O2	0.070 (3)	0.042 (2)	0.064 (3)	−0.007 (2)	0.012 (2)	0.0030 (18)
O3	0.040 (2)	0.063 (2)	0.068 (2)	−0.0066 (18)	0.0153 (18)	−0.008 (2)
N1	0.041 (2)	0.036 (2)	0.040 (2)	0.0023 (18)	0.0073 (17)	−0.0028 (17)
N2	0.044 (2)	0.041 (2)	0.044 (2)	0.002 (2)	0.0133 (19)	−0.0004 (19)
C1	0.042 (3)	0.050 (3)	0.039 (2)	−0.008 (2)	0.008 (2)	0.001 (2)
C2	0.038 (3)	0.047 (3)	0.050 (3)	0.002 (2)	0.007 (2)	−0.008 (2)
C3	0.044 (3)	0.037 (3)	0.056 (3)	0.004 (2)	0.007 (2)	−0.004 (2)
C4	0.042 (3)	0.030 (2)	0.041 (2)	−0.005 (2)	0.005 (2)	−0.0026 (19)

C5	0.038 (2)	0.037 (3)	0.035 (2)	−0.006 (2)	0.0018 (19)	−0.002 (2)
C6	0.045 (3)	0.035 (2)	0.042 (2)	0.002 (2)	0.005 (2)	0.0055 (19)
C7	0.043 (3)	0.029 (2)	0.041 (2)	0.001 (2)	0.004 (2)	−0.0008 (18)
C8	0.039 (3)	0.044 (3)	0.044 (2)	−0.004 (2)	0.015 (2)	0.0020 (19)
C9	0.046 (3)	0.043 (3)	0.048 (3)	0.004 (2)	0.005 (2)	0.004 (2)
C10	0.067 (4)	0.047 (3)	0.051 (3)	0.002 (3)	0.018 (3)	−0.007 (2)
C11	0.060 (4)	0.064 (4)	0.040 (3)	−0.014 (3)	0.011 (2)	0.001 (3)
C12	0.086 (6)	0.099 (6)	0.059 (4)	0.000 (5)	−0.008 (4)	−0.013 (4)
C13	0.047 (3)	0.077 (4)	0.055 (3)	0.011 (4)	0.005 (2)	0.011 (4)
C14	0.050 (3)	0.052 (3)	0.059 (3)	0.007 (3)	0.013 (3)	−0.003 (3)

Geometric parameters (Å, º)

I1—C1	2.092 (5)	C5—C7	1.447 (7)
S1—O2	1.418 (5)	C6—H6	0.9300
S1—O3	1.429 (4)	C7—H7	0.9300
S1—N2	1.640 (4)	C8—C9	1.379 (7)
S1—C8	1.774 (5)	C8—C14	1.384 (8)
O1—C4	1.364 (6)	C9—C10	1.381 (9)
O1—H1	0.82 (2)	C9—H9	0.9300
N1—C7	1.283 (6)	C10—C11	1.384 (9)
N1—N2	1.373 (6)	C10—H10	0.9300
N2—H2A	0.86 (2)	C11—C13	1.380 (11)
C1—C2	1.380 (8)	C11—C12	1.521 (11)
C1—C6	1.389 (7)	C12—H12A	0.9600
C2—C3	1.382 (8)	C12—H12B	0.9600
C2—H2	0.9300	C12—H12C	0.9600
C3—C4	1.385 (7)	C13—C14	1.371 (10)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.413 (7)	C14—H14	0.9300
C5—C6	1.390 (6)		
O2—S1—O3	121.6 (3)	C5—C6—H6	119.9
O2—S1—N2	106.4 (2)	N1—C7—C5	119.6 (4)
O3—S1—N2	104.6 (3)	N1—C7—H7	120.2
O2—S1—C8	108.7 (3)	C5—C7—H7	120.2
O3—S1—C8	108.4 (2)	C9—C8—C14	120.9 (5)
N2—S1—C8	106.1 (2)	C9—C8—S1	119.3 (4)
C4—O1—H1	111 (5)	C14—C8—S1	119.7 (4)
C7—N1—N2	119.3 (4)	C8—C9—C10	118.9 (6)
N1—N2—S1	115.6 (3)	C8—C9—H9	120.6
N1—N2—H2A	115 (5)	C10—C9—H9	120.6
S1—N2—H2A	120 (5)	C9—C10—C11	121.5 (6)
C2—C1—C6	120.9 (5)	C9—C10—H10	119.2
C2—C1—I1	119.2 (4)	C11—C10—H10	119.2
C6—C1—I1	119.8 (4)	C13—C11—C10	117.8 (6)
C1—C2—C3	119.7 (5)	C13—C11—C12	121.4 (7)
C1—C2—H2	120.1	C10—C11—C12	120.7 (7)

C3—C2—H2	120.1	C11—C12—H12A	109.5
C2—C3—C4	120.0 (5)	C11—C12—H12B	109.5
C2—C3—H3	120.0	H12A—C12—H12B	109.5
C4—C3—H3	120.0	C11—C12—H12C	109.5
O1—C4—C3	117.3 (5)	H12A—C12—H12C	109.5
O1—C4—C5	121.9 (4)	H12B—C12—H12C	109.5
C3—C4—C5	120.8 (5)	C14—C13—C11	122.1 (6)
C6—C5—C4	118.3 (4)	C14—C13—H13	118.9
C6—C5—C7	119.8 (5)	C11—C13—H13	118.9
C4—C5—C7	122.0 (4)	C13—C14—C8	118.7 (6)
C1—C6—C5	120.3 (5)	C13—C14—H14	120.6
C1—C6—H6	119.9	C8—C14—H14	120.6

C7—N1—N2—S1	−162.9 (4)	C6—C5—C7—N1	−174.8 (5)
O2—S1—N2—N1	−36.8 (5)	C4—C5—C7—N1	6.4 (7)
O3—S1—N2—N1	−166.7 (4)	O2—S1—C8—C9	−153.8 (4)
C8—S1—N2—N1	78.8 (4)	O3—S1—C8—C9	−19.8 (5)
C6—C1—C2—C3	−1.7 (8)	N2—S1—C8—C9	92.0 (4)
I1—C1—C2—C3	178.6 (4)	O2—S1—C8—C14	29.4 (5)
C1—C2—C3—C4	1.9 (8)	O3—S1—C8—C14	163.4 (4)
C2—C3—C4—O1	179.3 (5)	N2—S1—C8—C14	−84.8 (5)
C2—C3—C4—C5	−1.1 (8)	C14—C8—C9—C10	−1.5 (8)
O1—C4—C5—C6	179.7 (5)	S1—C8—C9—C10	−178.3 (4)
C3—C4—C5—C6	0.1 (7)	C8—C9—C10—C11	0.1 (8)
O1—C4—C5—C7	−1.5 (7)	C9—C10—C11—C13	0.9 (9)
C3—C4—C5—C7	179.0 (5)	C9—C10—C11—C12	178.4 (6)
C2—C1—C6—C5	0.7 (8)	C10—C11—C13—C14	−0.5 (9)
I1—C1—C6—C5	−179.6 (4)	C12—C11—C13—C14	−177.9 (7)
C4—C5—C6—C1	0.1 (7)	C11—C13—C14—C8	−0.9 (10)
C7—C5—C6—C1	−178.8 (5)	C9—C8—C14—C13	1.9 (8)
N2—N1—C7—C5	−173.7 (4)	S1—C8—C14—C13	178.7 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82 (2)	1.91 (5)	2.589 (6)	139 (7)
N2—H2*A*···O1^{i}	0.86 (2)	2.05 (2)	2.914 (6)	173 (6)

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