IUCrData

ISSN 2414-3146

N-(4-Chlorophenylsulfonyl)-4-iodobenzamide

S. Naveen,[a] A. G. Sudha,[b] E. Suresha,[b] N. K. Lokanath,[c] P. A. Suchetan[b]* and Ismail Warad[d]*

[a]Institution of Excellence, University of Mysore, Manasagangotri, Mysuru-6, India, [b]Department of Chemistry, University College of Science, Tumkur University, Tumkur 572 103, India, [c]Department of Studies in Physics, University of Mysore, Manasagangotri, Mysuru-6, India, and [d]Department of Chemistry, Science College, An-Najah National University, PO Box 7, Nablus, Palestinian Territories. *Correspondence e-mail: pasuchetan@yahoo.co.in, khalil.i@najah.edu

Received 1 January 2017
Accepted 6 January 2017

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

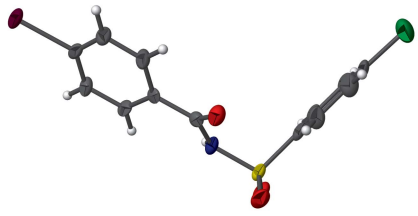
Keywords: crystal structure; sulfonamides; benzamides; *N*-(arylsulfonyl)arylamides; hydrogen bonding; offset π–π interactions.

CCDC reference: 1525994

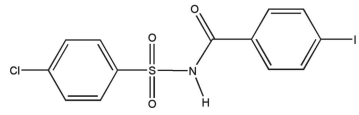
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_9ClINO_3S$, the benzene rings are inclined to one another by 81.6 (2)°. In the crystal, molecules are linked by pairs of N—H···O hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ ring motif. The dimers are linked by C—H···O hydrogen bonds, forming sheets parallel to the *bc* plane. Neighbouring sheets are linked *via* offset π–π interactions involving inversion-related iodobenzene rings [intercentroid distance = 3.807 (3) Å], forming a three-dimensional supramolecular structure.

3D view



Chemical scheme



Structure description

Sulfonamide and amide moieties play a significant role as key constituents in a number of biologically active molecules (Mohan *et al.*, 2013; Manojkumar *et al.*, 2013; Hamad & Abed, 2014). In recent years, *N*-(arylsulfonyl)-arylamides have received much attention as they constitute an important class of drugs for Alzheimer's disease (Hasegawa & Yamamoto, 2000), anti-bacterial inhibitors of tRNA synthetases (Banwell *et al.*, 2000), antagonists for angiotensin II (Chang *et al.*, 1994) and as leukotriene D4-receptors (Musser *et al.*, 1990). Further, *N*-(arylsulfonyl)-arylamides are known to be potent anti-tumour agents against a broad spectrum of human tumour xenografts (colon, lung, breast, ovary and prostate) in nude mice (Mader *et al.*, 2005). In view of the importance of *N*-(arylsulfonyl)-arylamides and in continuation of our work on the synthesis and crystal structures of *N*-(4-chlorophenylsulfonyl)-arylamides (Suchetan *et al.*, 2010*a*,*b*,*c*, 2011), the title compound was synthesized and we report herein on its crystal structure.

The molecular structure of the title compound is illustrated in Fig. 1. The molecule is V-shaped with the dihedral angle between the chlorobenzene and iodobenzene rings (C1–C6 and C8–C13, respectively) being 81.6 (2)°.

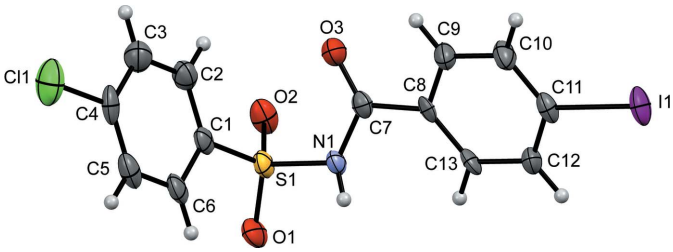


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

In the crystal, molecules are linked by pairs of N1—H*N*1···O1^{i} (Table 1) hydrogen bonds, forming an inversion dimer with an $R_2^2(8)$ ring motif. The dimers are linked *via* C12—H12···O3^{ii} (Table 1) hydrogen bonds, forming sheets parallel to the *bc* plane (Fig. 2). Neighbouring sheets are linked *via* offset π–π interactions involving inversion-related iodobenzene rings (*Cg* = centroid of ring C8–C13), forming a three-dimensional supramolecular structure, as illustrated in Fig. 3 [$Cg\cdots Cg^{iii}$ = 3.807 (3) Å; interplanar distance = 3.653 (2) Å; slippage 1.072 Å; symmetry code (iii) $-x+2, -y, -z+2$].

Synthesis and crystallization

The title compound was prepared by refluxing a mixture of 4-iodobenzoic acid (0.372 g, 1.5 mmol), 4-chlorobenzenesulfonamide (0.287 g, 1.5 mmol) and phosphorousoxychloride (7 ml) for 3 h on a water bath. The resultant mixture was cooled and poured into ice-cold water. The solid obtained was filtered, washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later re-precipitated by acidifying the filtered solution with dilute HCl. It was then filtered, dried and recrystallized from methanol

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H*N*1···O1^{i}	0.81 (4)	2.14 (4)	2.943 (5)	168 (6)
C12—H12···O3^{ii}	0.93	2.43	3.124 (6)	131

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_9ClINO_3S$
M_r	421.62
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	18.1163 (18), 13.3947 (13), 12.3002 (13)
β (°)	104.223 (4)
V (Å^3)	2893.3 (5)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	20.51
Crystal size (mm)	0.28 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.069, 0.080
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13818, 2369, 2178
R_{int}	0.072
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.172, 1.08
No. of reflections	2369
No. of parameters	185
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.58, −1.82

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

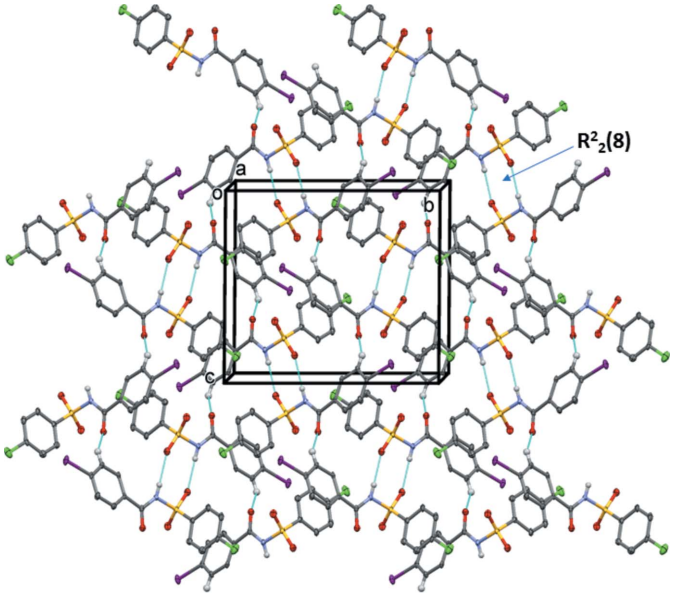


Figure 2
A view along the *a* axis of the crystal packing of the title compound, showing the N—H···O and C—H···O hydrogen bonds (see Table 1) as dashed lines. For clarity, only H atoms H*N*1 and H12 have been included.

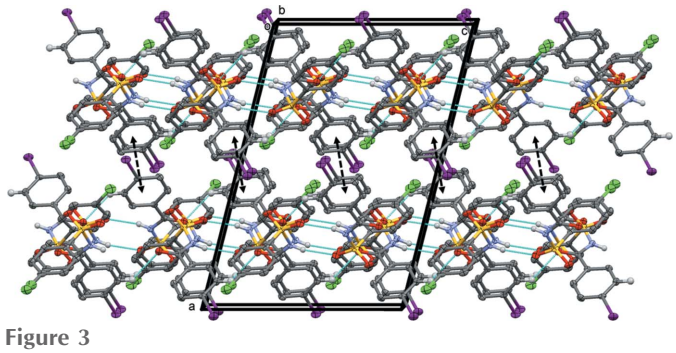


Figure 3
A view along the *b* axis of the crystal packing of the title compound, showing the N—H···O and C—H···O hydrogen bonds (dashed lines; see Table 1) and the π–π interactions (dashed arrows). For clarity, only H atoms H*N*1 and H12 have been included.

(m.p. 425 K). Colourless prismatic crystals were obtained by slow evaporation of a solution of the title compound in methanol (with a few drops of water).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysore, for providing the single-crystal X-ray diffraction data.

References

Banwell, M. G., Crasto, C. F., Easton, C. J., Forrest, A. K., Karoli, T., March, D. R., Mensah, L., Nairn, M. R., O'Hanlon, P. J., Oldham, M. D. & Yue, W. (2000). *Bioorg. Med. Chem. Lett.* **10**, 2263–2266.

Bruker (2009). *APEX2*, *SAINT-Plus*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chang, L. L., Ashton, W. T., Flanagan, K. L., Chen, T. B., O'Malley, S. S., Zingaro, G. J., Siegl, P. K. S., Kivlighn, S. D., Lotti, V. J. & Chang, R. S. L. (1994). *J. Med. Chem.* **37**, 4464–4478.

Hamad, A. S. & Abed, F. S. (2014). *J. Appl. Chem*, **3**, 56–63.

Hasegawa, T. & Yamamoto, H. (2000). *Bull. Chem. Soc. Jpn*, **73**, 423–428.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Mader, M., Shih, C., Considine, E., Dios, A. D., Grossman, C., Hipskind, P., Lin, H., Lobb, K., Lopez, B., Lopez, J., Cabrejas, L., Richett, M., White, W., Cheung, Y., Huang, Z., Reilly, J. & Dinn, S. (2005). *Bioorg. Med. Chem. Lett.* **15**, 617–620.

Manojkumar, K. E., Sreenivasa, S., Mohan, N. R., Madhu Chakrapani Rao, T. & Harikrishna, T. (2013). *J. Appl. Chem*, **2**, 730–737.

Mohan, N. R., Sreenivasa, S., Manojkumar, K. E. & Chakrapani Rao, T. M. (2013). *J. Appl. Chem*, **2**, 722–729.

Musser, J. H., Kreft, A. F., Bender, R. H. W., Kubrak, D. M., Grimes, D., Carlson, R. P., Hand, J. M. & Chang, J. (1990). *J. Med. Chem.* **33**, 240–245.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Suchetan, P. A., Foro, S. & Gowda, B. T. (2011). *Acta Cryst.* E**67**, o904.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*a*). *Acta Cryst.* E**66**, o766.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o1253.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*c*). *Acta Cryst.* E**66**, o1501.

full crystallographic data

IUCrData (2017). **2**, x170025 [https://doi.org/10.1107/S2414314617000256]

N-(4-Chlorophenylsulfonyl)-4-iodobenzamide

S. Naveen, A. G. Sudha, E. Suresha, N. K. Lokanath, P. A. Suchetan and Ismail Warad

N-(4-Chlorophenylsulfonyl)-4-iodobenzamide

Crystal data

$C_{13}H_9ClINO_3S$
M_r = 421.62
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 18.1163 (18) Å
b = 13.3947 (13) Å
c = 12.3002 (13) Å
β = 104.223 (4)°
V = 2893.3 (5) Å^3
Z = 8
F(000) = 1632
D_x = 1.936 Mg m^{-3}
Melting point: 425 K
Cu *K*α radiation, λ = 1.54178 Å
Cell parameters from 144 reflections
θ = 4.2–64.2°
μ = 20.51 mm^{-1}
T = 296 K
Prism, colourless
0.28 × 0.27 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.069, T_{max} = 0.080
13818 measured reflections
2369 independent reflections
2178 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 64.2°, θ_{min} = 4.2°
h = −21→20
k = −15→15
l = −12→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.172
S = 1.08
2369 reflections
185 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1376P)^2 + 0.079P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.58 e Å^{-3}
$\Delta\rho_{min}$ = −1.82 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
C1	0.7762 (2)	0.3242 (4)	0.7483 (4)	0.0274 (10)
C2	0.7821 (3)	0.3116 (4)	0.6400 (4)	0.0361 (11)
H2	0.7548	0.2611	0.5958	0.043*
C3	0.8284 (3)	0.3737 (5)	0.5966 (4)	0.0425 (12)
H3	0.8333	0.3650	0.5237	0.051*
C4	0.8676 (3)	0.4492 (4)	0.6636 (5)	0.0370 (12)
C5	0.8628 (3)	0.4624 (4)	0.7727 (5)	0.0391 (12)
H5	0.8902	0.5128	0.8168	0.047*
C6	0.8161 (3)	0.3987 (4)	0.8160 (5)	0.0336 (11)
H6	0.8119	0.4063	0.8894	0.040*
C7	0.8082 (3)	0.0884 (4)	0.8118 (4)	0.0244 (10)
C8	0.8589 (2)	0.0109 (3)	0.8799 (4)	0.0220 (9)
C9	0.9104 (3)	−0.0367 (4)	0.8302 (4)	0.0319 (11)
H9	0.9131	−0.0180	0.7584	0.038*
C10	0.9575 (3)	−0.1108 (4)	0.8851 (4)	0.0334 (11)
H10	0.9926	−0.1413	0.8519	0.040*
C11	0.9518 (2)	−0.1392 (4)	0.9901 (4)	0.0268 (10)
C12	0.9013 (3)	−0.0940 (4)	1.0412 (4)	0.0295 (10)
H12	0.8981	−0.1142	1.1122	0.035*
C13	0.8553 (3)	−0.0179 (3)	0.9861 (4)	0.0264 (10)
H13	0.8217	0.0139	1.0210	0.032*
O1	0.69380 (19)	0.2958 (3)	0.8916 (3)	0.0374 (8)
O2	0.66080 (18)	0.2040 (3)	0.7129 (3)	0.0377 (8)
O3	0.79821 (18)	0.0931 (3)	0.7109 (3)	0.0340 (8)
S1	0.71643 (8)	0.24483 (8)	0.80304 (11)	0.0264 (4)
Cl1	0.92287 (8)	0.52981 (13)	0.60795 (14)	0.0601 (5)
I1	1.02099 (2)	−0.25581 (3)	1.07348 (3)	0.0422 (3)
N1	0.7722 (2)	0.1537 (3)	0.8684 (3)	0.0251 (8)
HN1	0.788 (3)	0.169 (4)	0.934 (3)	0.023 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.029 (2)	0.018 (2)	0.030 (2)	0.0099 (18)	−0.0016 (17)	0.0023 (18)
C2	0.044 (3)	0.025 (3)	0.034 (2)	−0.003 (2)	0.0003 (19)	0.000 (2)
C3	0.052 (3)	0.034 (3)	0.039 (3)	0.000 (2)	0.007 (2)	0.003 (2)
C4	0.031 (2)	0.020 (3)	0.055 (3)	0.005 (2)	0.002 (2)	0.017 (2)
C5	0.037 (2)	0.018 (3)	0.054 (3)	0.004 (2)	−0.003 (2)	0.002 (2)
C6	0.039 (3)	0.016 (3)	0.042 (3)	0.010 (2)	0.003 (2)	0.001 (2)
C7	0.023 (2)	0.017 (3)	0.032 (2)	−0.0035 (18)	0.0049 (17)	−0.0002 (19)

C8	0.024 (2)	0.008 (2)	0.033 (2)	−0.0047 (16)	0.0054 (16)	−0.0032 (17)
C9	0.043 (3)	0.021 (3)	0.035 (2)	0.007 (2)	0.0149 (19)	0.0056 (19)
C10	0.037 (2)	0.023 (3)	0.044 (3)	0.010 (2)	0.017 (2)	0.005 (2)
C11	0.0203 (19)	0.015 (2)	0.041 (2)	0.0018 (17)	0.0004 (17)	−0.0008 (18)
C12	0.039 (2)	0.018 (2)	0.030 (2)	0.0058 (19)	0.0069 (18)	0.0031 (18)
C13	0.029 (2)	0.013 (2)	0.038 (2)	0.0068 (18)	0.0107 (17)	−0.0039 (18)
O1	0.0419 (19)	0.029 (2)	0.0407 (18)	0.0185 (16)	0.0087 (14)	0.0006 (16)
O2	0.0268 (16)	0.030 (2)	0.0481 (19)	0.0005 (15)	−0.0071 (14)	−0.0011 (17)
O3	0.0409 (17)	0.028 (2)	0.0330 (17)	0.0073 (14)	0.0079 (13)	0.0012 (14)
S1	0.0267 (8)	0.0184 (7)	0.0315 (7)	0.0071 (4)	0.0020 (5)	0.0020 (4)
Cl1	0.0554 (9)	0.0455 (9)	0.0771 (10)	−0.0123 (7)	0.0119 (7)	0.0213 (7)
I1	0.0384 (4)	0.0233 (4)	0.0621 (4)	0.01269 (11)	0.0068 (3)	0.01117 (13)
N1	0.0307 (19)	0.016 (2)	0.0258 (19)	0.0058 (16)	0.0024 (14)	−0.0003 (16)

Geometric parameters (Å, º)

C1—C2	1.374 (7)	C8—C13	1.379 (7)
C1—C6	1.385 (8)	C8—C9	1.390 (6)
C1—S1	1.764 (5)	C9—C10	1.374 (7)
C2—C3	1.379 (8)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.375 (7)
C3—C4	1.385 (8)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.370 (7)
C4—C5	1.377 (8)	C11—I1	2.105 (4)
C4—Cl1	1.725 (5)	C12—C13	1.384 (7)
C5—C6	1.395 (8)	C12—H12	0.9300
C5—H5	0.9300	C13—H13	0.9300
C6—H6	0.9300	O1—S1	1.429 (4)
C7—O3	1.211 (6)	O2—S1	1.413 (4)
C7—N1	1.379 (6)	S1—N1	1.661 (4)
C7—C8	1.499 (7)	N1—HN1	0.81 (3)
C2—C1—C6	121.1 (5)	C10—C9—C8	121.2 (4)
C2—C1—S1	119.9 (4)	C10—C9—H9	119.4
C6—C1—S1	119.0 (4)	C8—C9—H9	119.4
C1—C2—C3	120.1 (5)	C9—C10—C11	118.7 (4)
C1—C2—H2	119.9	C9—C10—H10	120.6
C3—C2—H2	119.9	C11—C10—H10	120.6
C2—C3—C4	118.8 (5)	C12—C11—C10	121.5 (4)
C2—C3—H3	120.6	C12—C11—I1	119.3 (3)
C4—C3—H3	120.6	C10—C11—I1	119.3 (3)
C5—C4—C3	121.9 (5)	C11—C12—C13	119.4 (4)
C5—C4—Cl1	119.2 (4)	C11—C12—H12	120.3
C3—C4—Cl1	118.9 (4)	C13—C12—H12	120.3
C4—C5—C6	118.8 (5)	C8—C13—C12	120.4 (4)
C4—C5—H5	120.6	C8—C13—H13	119.8
C6—C5—H5	120.6	C12—C13—H13	119.8
C1—C6—C5	119.2 (5)	O2—S1—O1	120.0 (2)

C1—C6—H6	120.4	O2—S1—N1	109.1 (2)
C5—C6—H6	120.4	O1—S1—N1	103.7 (2)
O3—C7—N1	121.0 (4)	O2—S1—C1	108.6 (2)
O3—C7—C8	121.7 (4)	O1—S1—C1	108.7 (2)
N1—C7—C8	117.2 (4)	N1—S1—C1	105.7 (2)
C13—C8—C9	118.8 (4)	C7—N1—S1	121.9 (3)
C13—C8—C7	124.0 (4)	C7—N1—HN1	125 (4)
C9—C8—C7	117.2 (4)	S1—N1—HN1	109 (4)
C6—C1—C2—C3	0.0 (7)	C9—C10—C11—I1	178.1 (4)
S1—C1—C2—C3	179.7 (4)	C10—C11—C12—C13	−0.2 (7)
C1—C2—C3—C4	−0.9 (8)	I1—C11—C12—C13	−179.4 (3)
C2—C3—C4—C5	1.5 (8)	C9—C8—C13—C12	−1.0 (7)
C2—C3—C4—Cl1	−177.7 (4)	C7—C8—C13—C12	176.5 (4)
C3—C4—C5—C6	−1.1 (7)	C11—C12—C13—C8	1.3 (7)
Cl1—C4—C5—C6	178.1 (4)	C2—C1—S1—O2	−22.7 (4)
C2—C1—C6—C5	0.4 (7)	C6—C1—S1—O2	156.9 (4)
S1—C1—C6—C5	−179.3 (4)	C2—C1—S1—O1	−155.0 (4)
C4—C5—C6—C1	0.2 (7)	C6—C1—S1—O1	24.7 (4)
O3—C7—C8—C13	−159.7 (5)	C2—C1—S1—N1	94.3 (4)
N1—C7—C8—C13	19.3 (6)	C6—C1—S1—N1	−86.1 (4)
O3—C7—C8—C9	17.9 (7)	O3—C7—N1—S1	−2.7 (6)
N1—C7—C8—C9	−163.1 (4)	C8—C7—N1—S1	178.3 (3)
C13—C8—C9—C10	−0.3 (7)	O2—S1—N1—C7	53.2 (4)
C7—C8—C9—C10	−178.0 (5)	O1—S1—N1—C7	−177.7 (4)
C8—C9—C10—C11	1.4 (8)	C1—S1—N1—C7	−63.5 (4)
C9—C10—C11—C12	−1.1 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H*N*1···O1^{i}	0.81 (4)	2.14 (4)	2.943 (5)	168 (6)
C12—H12···O3^{ii}	0.93	2.43	3.124 (6)	131

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