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N-(4-Chlorophenyl)-2-nitrobenzenesulfonamide

 U. Chaithanya,^a Sabine Foro^b and B. Thimme Gowda^{a*}
^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

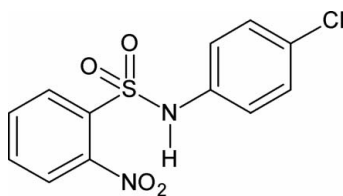
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 14.8.

In the title compound, $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$, the dihedral angle between the aromatic rings is $70.27(8)^\circ$. In the crystal, molecules are linked by pairs of $\text{N}-\text{H}\cdots\text{O}(\text{S})$ hydrogen bonds, forming inversion dimers.

Related literature

For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Alkan *et al.* (2011); Bowes *et al.* (2003); Gowda *et al.* (2000); Saeed *et al.* (2010); Shahwar *et al.* (2012), of *N*-aroylsulfonamides, see: Chaithanya *et al.* (2012), of *N*-arylsulfonamides, see: Gowda *et al.* (2002) and of *N*-chloroarylsulfonamides, see: Gowda & Shetty (2004); Shetty & Gowda (2004).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$
 $M_r = 312.72$

 Monoclinic, $P2_1/c$
 $a = 8.3295(4)$ Å

 $b = 11.0866(7)$ Å

 $c = 14.5576(8)$ Å

 $\beta = 92.481(5)^\circ$
 $V = 1343.07(13)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.45$ mm⁻¹
 $T = 293$ K

 $0.44 \times 0.40 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)

 $T_{\min} = 0.825$, $T_{\max} = 0.915$

5440 measured reflections

2731 independent reflections

 2314 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.096$
 $S = 1.04$

2731 reflections

185 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.85 (1)	2.27 (1)	3.084 (2)	161 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5983).

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supporting information

Acta Cryst. (2012). E68, o2577 [https://doi.org/10.1107/S1600536812033429]

N-(4-Chlorophenyl)-2-nitrobenzenesulfonamide

U. Chaithanya, Sabine Foro and B. Thimme Gowda

S1. Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Alkan *et al.*, 2011; Bowes *et al.*, 2003; Gowda *et al.*, 2000; Saeed *et al.*, 2010; Shahwar *et al.*, 2012); *N*-arylsulfonamides (Chaithanya *et al.*, 2012); *N*-arylsulfonamides (Gowda *et al.*, 2002) and *N*-chloroarylsulfonamides (Gowda & Shetty, 2004; Shetty & Gowda, 2004), in the present work, the crystal structure of *N*-(4-chlorophenyl)-2-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—H bond in the —SO₂—NH— segment is *syn* to the *ortho*-nitro group in the sulfonyl benzene ring, similar to that observed in *N*-(phenyl)-2-nitrobenzenesulfonamide (I) (Chaithanya *et al.*, 2012). The molecule is twisted at the S—N bond with the torsional angle of 79.17 (18)°, compared to the value of -72.83 (15)° in (I).

The dihedral angle between the sulfonyl and the anilino rings is 70.27 (8)°, compared to the value of 59.55 (7)° in (I).

In the crystal, the pairs of intermolecular N—H...O (S) hydrogen bonds (Table 1) link the molecules into inversion dimers. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The title compound was prepared by treating 2-nitrobenzenesulfonylchloride with 4-chloroaniline in the stoichiometric ratio and boiling the reaction mixture for 15 minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid *N*-(4-chlorophenyl)-2-nitrobenzenesulfonamide was filtered under suction and washed thoroughly with cold water and dilute HCl to remove the excess sulfonylchloride and aniline, respectively. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like yellow single crystals of the title compound used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

S3. Refinement

H atoms bonded to C were positioned with idealized geometry using a riding model with C—H = 0.93 Å. The coordinates of the amino H atom were refined with the N—H distance restrained to 0.86 (1) Å. All H atoms were refined with isotropic displacement parameters set to 1.2 U_{eq} of the parent atom. The (0 1 2 and 0 3 3) reflections were probably affected by the beamstop and were omitted from the refinement.

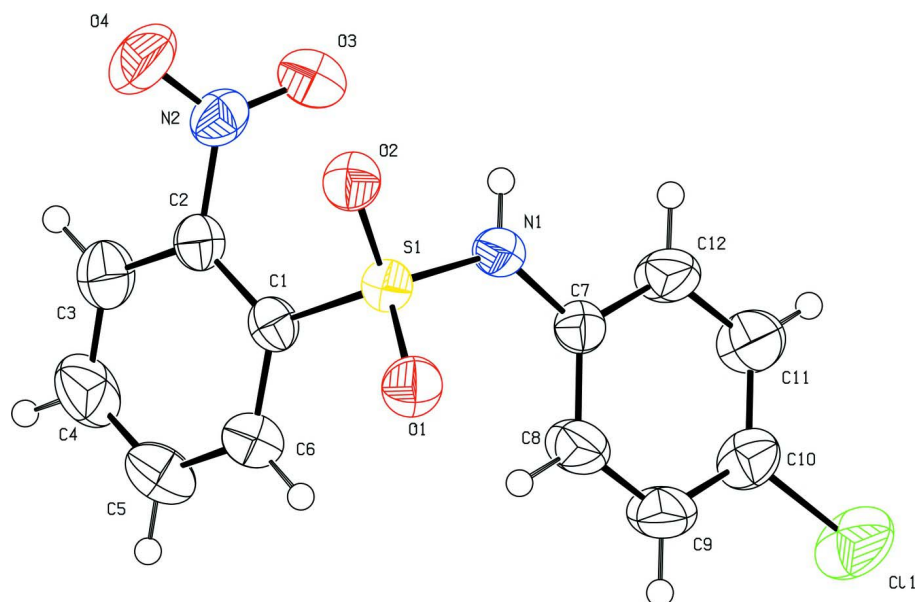


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

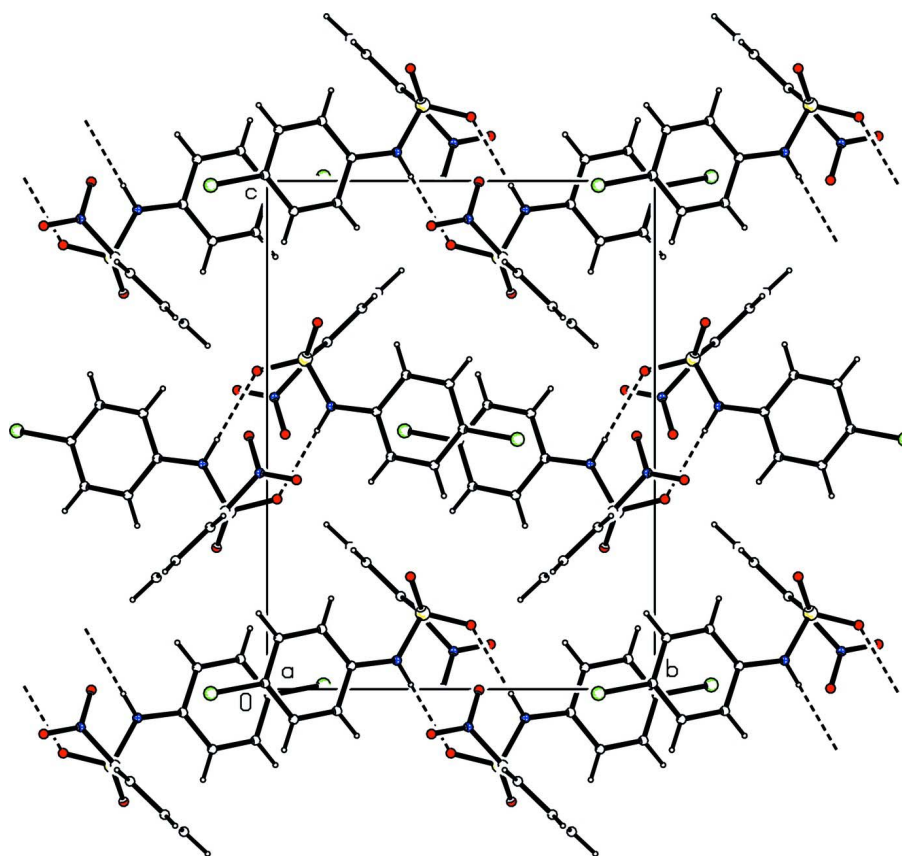


Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(4-Chlorophenyl)-2-nitrobenzenesulfonamide

Crystal data

$C_{12}H_9ClN_2O_4S$

$M_r = 312.72$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.3295$ (4) Å

$b = 11.0866$ (7) Å

$c = 14.5576$ (8) Å

$\beta = 92.481$ (5)°

$V = 1343.07$ (13) Å³

$Z = 4$

$F(000) = 640$

$D_x = 1.547$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2996 reflections

$\theta = 2.9$ – 27.8 °

$\mu = 0.45$ mm⁻¹

$T = 293$ K

Prism, yellow

$0.44 \times 0.40 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Rotation method data acquisition using ω scans

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.825$, $T_{\max} = 0.915$

5440 measured reflections

2731 independent reflections

2314 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 3.3$ °

$h = -7 \rightarrow 10$

$k = -5 \rightarrow 13$

$l = -18 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.096$

$S = 1.03$

2731 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

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.5971P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.30$ e Å⁻³

$\Delta\rho_{\min} = -0.33$ e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.073 (3)

Special details

Experimental. Absorption correction: *CrysAlis RED* (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in *SCALE3 ABSPACK* scaling algorithm.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1798 (2)	0.15532 (16)	0.68296 (11)	0.0370 (4)
C2	-0.3256 (2)	0.10822 (17)	0.64898 (12)	0.0402 (4)
C3	-0.4701 (2)	0.1467 (2)	0.68128 (16)	0.0573 (5)
H3	-0.5663	0.1128	0.6590	0.069*
C4	-0.4699 (3)	0.2359 (3)	0.74690 (18)	0.0693 (7)
H4	-0.5668	0.2630	0.7688	0.083*
C5	-0.3281 (3)	0.2855 (2)	0.78045 (16)	0.0637 (6)
H5	-0.3295	0.3468	0.8241	0.076*
C6	-0.1826 (2)	0.24446 (19)	0.74942 (13)	0.0487 (5)
H6	-0.0868	0.2770	0.7734	0.058*
C7	0.1198 (2)	0.28269 (16)	0.54582 (12)	0.0376 (4)
C8	0.1634 (3)	0.3588 (2)	0.61813 (15)	0.0620 (6)
H8	0.1483	0.3348	0.6784	0.074*
C9	0.2294 (3)	0.4702 (2)	0.60068 (16)	0.0642 (6)
H9	0.2577	0.5216	0.6492	0.077*
C10	0.2532 (3)	0.50542 (18)	0.51187 (15)	0.0511 (5)
C11	0.2103 (3)	0.4313 (2)	0.44058 (15)	0.0668 (7)
H11	0.2248	0.4560	0.3804	0.080*
C12	0.1453 (3)	0.3197 (2)	0.45749 (14)	0.0570 (5)
H12	0.1182	0.2686	0.4085	0.068*
N1	0.0544 (2)	0.16573 (15)	0.55669 (10)	0.0424 (4)
H1N	0.019 (3)	0.1292 (18)	0.5090 (10)	0.051*
N2	-0.33334 (18)	0.01897 (15)	0.57452 (12)	0.0462 (4)
O1	0.12301 (16)	0.13015 (13)	0.72132 (9)	0.0496 (4)
O2	-0.01505 (15)	-0.02583 (11)	0.62602 (9)	0.0449 (3)
O3	-0.27587 (19)	0.04674 (15)	0.50164 (10)	0.0596 (4)
O4	-0.4002 (2)	-0.07600 (16)	0.58813 (15)	0.0779 (5)
Cl1	0.33847 (10)	0.64535 (6)	0.49159 (5)	0.0812 (3)
S1	0.00868 (5)	0.09845 (4)	0.64966 (3)	0.03638 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0395 (9)	0.0376 (9)	0.0342 (8)	0.0032 (7)	0.0038 (7)	0.0049 (7)
C2	0.0402 (9)	0.0407 (9)	0.0400 (9)	0.0001 (7)	0.0041 (7)	0.0040 (8)
C3	0.0405 (10)	0.0664 (14)	0.0658 (13)	0.0030 (10)	0.0088 (9)	-0.0012 (11)
C4	0.0541 (13)	0.0838 (18)	0.0713 (15)	0.0174 (12)	0.0189 (11)	-0.0103 (13)
C5	0.0712 (14)	0.0654 (15)	0.0555 (13)	0.0134 (12)	0.0126 (11)	-0.0142 (11)
C6	0.0534 (11)	0.0507 (12)	0.0418 (10)	0.0028 (9)	0.0011 (8)	-0.0050 (9)
C7	0.0377 (8)	0.0363 (9)	0.0391 (9)	0.0011 (7)	0.0032 (7)	0.0007 (7)
C8	0.0986 (17)	0.0488 (12)	0.0398 (10)	-0.0157 (12)	0.0155 (11)	-0.0041 (9)
C9	0.0994 (18)	0.0435 (12)	0.0503 (12)	-0.0164 (12)	0.0088 (12)	-0.0097 (10)
C10	0.0600 (12)	0.0373 (10)	0.0556 (12)	-0.0039 (9)	-0.0025 (9)	0.0079 (9)
C11	0.0955 (18)	0.0649 (15)	0.0395 (11)	-0.0244 (13)	-0.0053 (11)	0.0119 (10)
C12	0.0789 (14)	0.0545 (12)	0.0369 (10)	-0.0184 (11)	-0.0052 (9)	0.0008 (9)

N1	0.0528 (9)	0.0405 (9)	0.0342 (8)	-0.0085 (7)	0.0060 (7)	-0.0035 (7)
N2	0.0363 (8)	0.0445 (9)	0.0574 (10)	-0.0011 (7)	-0.0027 (7)	-0.0034 (8)
O1	0.0440 (7)	0.0604 (9)	0.0438 (7)	-0.0017 (6)	-0.0070 (6)	0.0037 (6)
O2	0.0472 (7)	0.0353 (7)	0.0524 (8)	0.0035 (5)	0.0041 (6)	0.0027 (6)
O3	0.0684 (9)	0.0651 (10)	0.0452 (8)	0.0018 (8)	0.0016 (7)	-0.0075 (7)
O4	0.0727 (11)	0.0533 (10)	0.1085 (15)	-0.0248 (9)	0.0137 (10)	-0.0102 (10)
Cl1	0.1132 (6)	0.0494 (4)	0.0801 (4)	-0.0267 (3)	-0.0066 (4)	0.0173 (3)
S1	0.0362 (2)	0.0365 (3)	0.0364 (2)	0.00091 (17)	0.00049 (16)	0.00247 (17)

Geometric parameters (Å, °)

C1—C6	1.384 (3)	C8—C9	1.380 (3)
C1—C2	1.393 (3)	C8—H8	0.9300
C1—S1	1.7782 (17)	C9—C10	1.373 (3)
C2—C3	1.379 (3)	C9—H9	0.9300
C2—N2	1.467 (2)	C10—C11	1.359 (3)
C3—C4	1.375 (3)	C10—Cl1	1.737 (2)
C3—H3	0.9300	C11—C12	1.378 (3)
C4—C5	1.374 (4)	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.388 (3)	N1—S1	1.6056 (15)
C5—H5	0.9300	N1—H1N	0.845 (10)
C6—H6	0.9300	N2—O4	1.212 (2)
C7—C12	1.375 (3)	N2—O3	1.222 (2)
C7—C8	1.385 (3)	O1—S1	1.4258 (14)
C7—N1	1.418 (2)	O2—S1	1.4318 (14)
C6—C1—C2	118.34 (17)	C10—C9—C8	120.2 (2)
C6—C1—S1	119.07 (14)	C10—C9—H9	119.9
C2—C1—S1	122.46 (14)	C8—C9—H9	119.9
C3—C2—C1	121.58 (19)	C11—C10—C9	120.2 (2)
C3—C2—N2	116.70 (17)	C11—C10—Cl1	120.40 (17)
C1—C2—N2	121.67 (16)	C9—C10—Cl1	119.40 (17)
C4—C3—C2	119.0 (2)	C10—C11—C12	119.9 (2)
C4—C3—H3	120.5	C10—C11—H11	120.0
C2—C3—H3	120.5	C12—C11—H11	120.0
C5—C4—C3	120.7 (2)	C7—C12—C11	120.8 (2)
C5—C4—H4	119.7	C7—C12—H12	119.6
C3—C4—H4	119.7	C11—C12—H12	119.6
C4—C5—C6	120.2 (2)	C7—N1—S1	128.70 (13)
C4—C5—H5	119.9	C7—N1—H1N	117.9 (16)
C6—C5—H5	119.9	S1—N1—H1N	112.6 (16)
C1—C6—C5	120.2 (2)	O4—N2—O3	124.13 (19)
C1—C6—H6	119.9	O4—N2—C2	118.12 (18)
C5—C6—H6	119.9	O3—N2—C2	117.71 (16)
C12—C7—C8	118.91 (18)	O1—S1—O2	119.60 (8)
C12—C7—N1	116.91 (17)	O1—S1—N1	109.08 (8)
C8—C7—N1	124.15 (17)	O2—S1—N1	106.25 (8)

C9—C8—C7	119.90 (19)	O1—S1—C1	106.42 (8)
C9—C8—H8	120.0	O2—S1—C1	107.00 (8)
C7—C8—H8	120.0	N1—S1—C1	108.04 (8)
C6—C1—C2—C3	-1.3 (3)	C8—C7—C12—C11	-1.2 (4)
S1—C1—C2—C3	174.54 (16)	N1—C7—C12—C11	-179.2 (2)
C6—C1—C2—N2	176.19 (17)	C10—C11—C12—C7	1.3 (4)
S1—C1—C2—N2	-8.0 (2)	C12—C7—N1—S1	-178.85 (16)
C1—C2—C3—C4	1.7 (3)	C8—C7—N1—S1	3.3 (3)
N2—C2—C3—C4	-175.9 (2)	C3—C2—N2—O4	-58.4 (3)
C2—C3—C4—C5	-0.5 (4)	C1—C2—N2—O4	124.1 (2)
C3—C4—C5—C6	-1.1 (4)	C3—C2—N2—O3	119.5 (2)
C2—C1—C6—C5	-0.3 (3)	C1—C2—N2—O3	-58.1 (2)
S1—C1—C6—C5	-176.30 (17)	C7—N1—S1—O1	-36.12 (19)
C4—C5—C6—C1	1.5 (4)	C7—N1—S1—O2	-166.30 (16)
C12—C7—C8—C9	0.8 (4)	C7—N1—S1—C1	79.17 (18)
N1—C7—C8—C9	178.6 (2)	C6—C1—S1—O1	18.79 (17)
C7—C8—C9—C10	-0.6 (4)	C2—C1—S1—O1	-157.00 (15)
C8—C9—C10—C11	0.7 (4)	C6—C1—S1—O2	147.73 (15)
C8—C9—C10—C11	-179.3 (2)	C2—C1—S1—O2	-28.05 (17)
C9—C10—C11—C12	-1.1 (4)	C6—C1—S1—N1	-98.23 (16)
C11—C10—C11—C12	178.9 (2)	C2—C1—S1—N1	85.98 (16)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O2 ⁱ	0.85 (1)	2.27 (1)	3.084 (2)	161 (2)

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