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N-(2-Chlorophenyl)-2-nitrobenzene-sulfonamide

 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

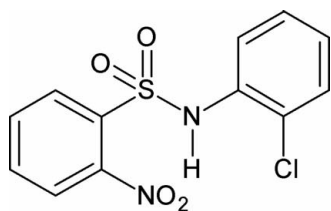
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.098; data-to-parameter ratio = 15.5.

In the title compound, $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$, the N—H bond in the $-\text{SO}_2-\text{NH}-$ segment is *syn* to both the *ortho*-nitro group in the sulfonylbenzene ring and the *ortho*-Cl atom in the aniline ring. The molecule is twisted at the S—N bond with a torsion angle of $75.0(2)^\circ$. The dihedral angle between the sulfonylbenzene and aniline rings is $54.97(11)^\circ$. The amide H atom shows bifurcated hydrogen bonding, generating $S(7)$ and $C(4)$ motifs. In the crystal, N—H \cdots O(S) hydrogen bonds link the molecules into chains.

Related literature

For studies of 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: Suchetan *et al.* (2012), of *N*-chloroarylsulfonamides, see: Gowda *et al.* (2005); Shetty & Gowda (2004) and of *N*-bromoaryl-sulfonamides, see: Gowda & Mahadevappa (1983); Usha & Gowda (2006). For hydrogen-bonding patterns and motifs, see: Admond & Grant (2001); Allen *et al.* (1998); Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$
 $M_r = 312.72$
 Monoclinic, $P2_1/c$
 $a = 9.2477(9)$ Å
 $b = 15.293(1)$ Å

 $c = 10.4671(9)$ Å
 $\beta = 108.66(1)^\circ$
 $V = 1402.5(2)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.43$ mm⁻¹
 $T = 293$ K

 $0.40 \times 0.32 \times 0.16$ mm

Data collection

 Oxford Diffraction Xcalibur
 diffractometer with a Sapphire
 CCD detector
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford)

 Diffraction, 2009)
 $T_{\min} = 0.845$, $T_{\max} = 0.934$
 5456 measured reflections
 2847 independent reflections
 2331 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.098$
 $S = 1.13$
 2847 reflections
 184 parameters
 1 restraint

 H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^1$	0.84 (2)	2.17 (2)	2.844 (2)	138 (2)
$\text{N1}-\text{H1N}\cdots\text{O3}$	0.84 (2)	2.49 (2)	3.099 (3)	130 (2)

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

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: SJ5261).

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N-(2-Chlorophenyl)-2-nitrobenzenesulfonamide

U. Chaithanya, Sabine Foro and B. Thimme Gowda

S1. Comment

As part of our studies of 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 (Suchetan *et al.*, 2012); *N*-chloroarylsulfonamides (Gowda *et al.*, 2005; Shetty & Gowda, 2004) and *N*-bromoarylsulfonamides (Gowda & Mahadevappa, 1983; Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(2-Chlorophenyl)-2-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—H bond in the —SO₂—NH— segment is *syn* to both the *ortho*-nitro group in the sulfonyl benzene ring and *ortho*-Cl atom in the anilino ring, similar to that observed in *N*-(2-chlorobenzoyl)-2-nitrobenzenesulfonamide (I) (Suchetan *et al.*, 2012). The molecule is twisted at the S—N bond with the torsional angle of 74.97 (20)°, compared to the value of -59.68 (17)° in (I).

The dihedral angle between the sulfonyl and the anilino rings is 54.97 (11)°, compared to the value of 71.2 (1)° in (I).

The amide H-atom shows bifurcated intramolecular H-bonding with the O-atom of the *ortho*-nitro group in the sulfonyl benzene ring and the intermolecular H-bonding with the sulfonyl oxygen atom of the other molecule, generating S(7) and C(4) motifs (Adsmund & Grant 2001; Allen *et al.*, 1998; Bernstein *et al.*, 1995; Etter, 1990).

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

S2. Experimental

The title compound was prepared by treating 2-nitrobenzenesulfonylchloride with 2-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 resulting solid *N*-(2-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 (145° C) from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like colourless 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 the aromatic C—H = 0.93 Å. The amino H atom was freely refined with the N—H distances restrained to 0.86 (2) Å. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq} of the parent atom.

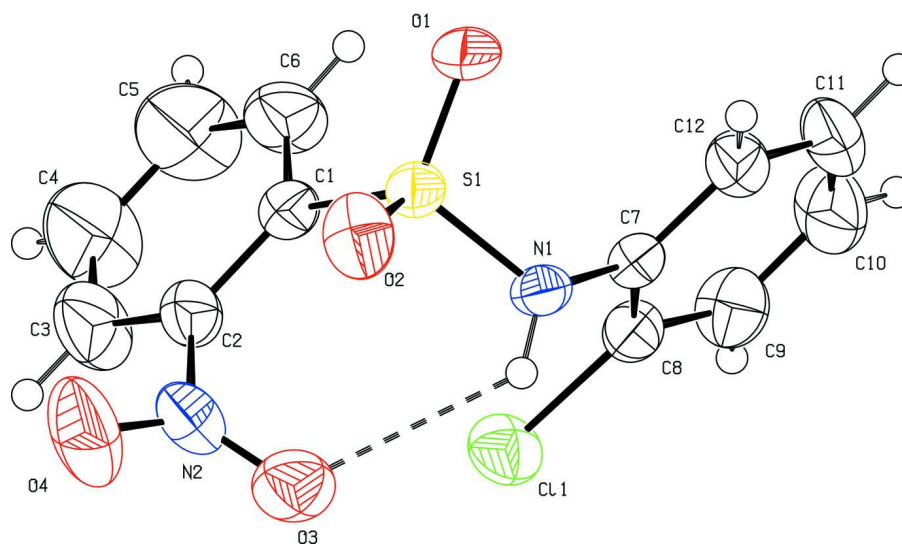


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level. An intramolecular hydrogen bond is drawn as a dashed line.

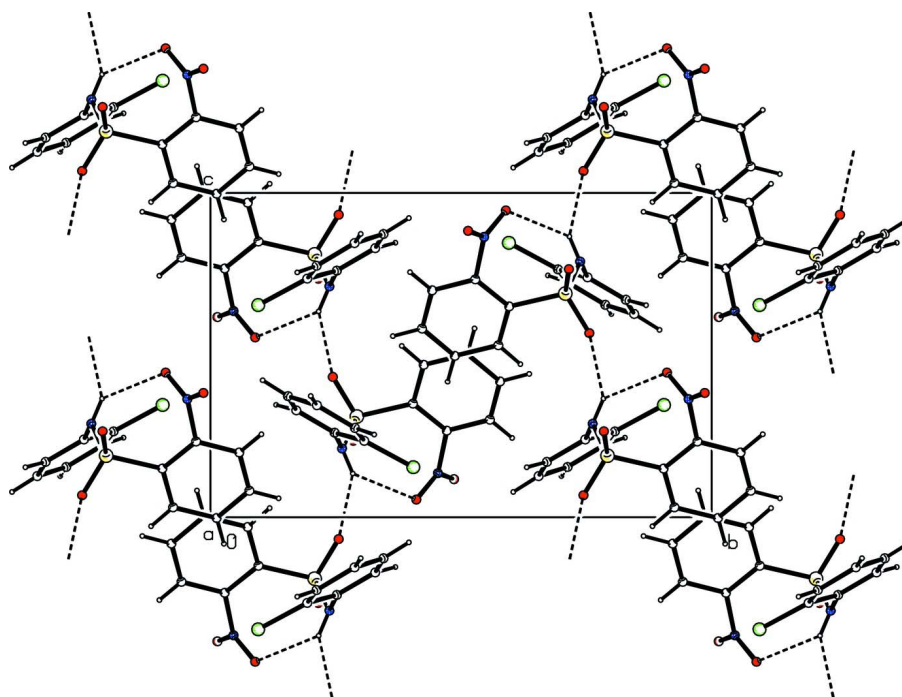


Figure 2

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

N-(2-Chlorophenyl)-2-nitrobenzenesulfonamide

Crystal data

$C_{12}H_9ClN_2O_4S$

$M_r = 312.72$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 9.2477\ (9)\ \text{\AA}$

$b = 15.293\ (1)\ \text{\AA}$

$c = 10.4671$ (9) Å
 $\beta = 108.66$ (1)°
 $V = 1402.5$ (2) Å³
 $Z = 4$
 $F(000) = 640$
 $D_x = 1.481$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2659 reflections

$\theta = 2.6$ – 27.9 °
 $\mu = 0.43$ mm⁻¹
 $T = 293$ K
 Prism, colourless
 $0.40 \times 0.32 \times 0.16$ 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)
 $T_{\min} = 0.845$, $T_{\max} = 0.934$

5456 measured reflections
 2847 independent reflections
 2331 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$
 $\theta_{\text{max}} = 26.4$ °, $\theta_{\text{min}} = 2.7$ °
 $h = -10 \rightarrow 11$
 $k = -10 \rightarrow 19$
 $l = -13 \rightarrow 8$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.098$
 $S = 1.13$
 2847 reflections
 184 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
 $w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 1.0512P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3704 (3)	0.90407 (15)	0.1498 (2)	0.0399 (5)
C2	0.4480 (3)	0.97153 (16)	0.2333 (3)	0.0453 (6)
C3	0.4168 (4)	1.0576 (2)	0.1994 (4)	0.0742 (10)
H3	0.4712	1.1017	0.2556	0.089*
C4	0.3045 (5)	1.0777 (2)	0.0816 (4)	0.1019 (15)
H4	0.2812	1.1360	0.0583	0.122*
C5	0.2257 (5)	1.0125 (3)	-0.0026 (4)	0.0996 (14)
H5	0.1492	1.0269	-0.0821	0.120*
C6	0.2596 (4)	0.9256 (2)	0.0303 (3)	0.0654 (8)

H6	0.2078	0.8817	-0.0280	0.079*
C7	0.1554 (2)	0.74776 (15)	0.2396 (2)	0.0339 (5)
C8	0.0533 (3)	0.80681 (15)	0.2644 (2)	0.0393 (5)
C9	-0.1011 (3)	0.7882 (2)	0.2248 (3)	0.0555 (7)
H9	-0.1683	0.8267	0.2453	0.067*
C10	-0.1544 (3)	0.7124 (2)	0.1549 (3)	0.0667 (8)
H10	-0.2584	0.7002	0.1266	0.080*
C11	-0.0550 (3)	0.6547 (2)	0.1267 (3)	0.0621 (8)
H11	-0.0922	0.6042	0.0777	0.075*
C12	0.0997 (3)	0.67125 (17)	0.1705 (3)	0.0469 (6)
H12	0.1667	0.6310	0.1537	0.056*
N1	0.3166 (2)	0.76256 (13)	0.28819 (18)	0.0343 (4)
H1N	0.353 (3)	0.7839 (15)	0.3657 (18)	0.041*
N2	0.5643 (3)	0.95397 (14)	0.3642 (2)	0.0514 (6)
O1	0.3492 (2)	0.74417 (12)	0.06758 (17)	0.0559 (5)
O2	0.56839 (19)	0.78488 (12)	0.2647 (2)	0.0550 (5)
O3	0.5276 (2)	0.91073 (14)	0.44578 (19)	0.0627 (5)
O4	0.6898 (3)	0.98600 (16)	0.3829 (3)	0.0893 (8)
Cl1	0.11837 (8)	0.90467 (4)	0.34591 (7)	0.05248 (19)
S1	0.41059 (7)	0.79209 (4)	0.18966 (6)	0.03658 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0484 (13)	0.0379 (12)	0.0350 (12)	-0.0023 (11)	0.0155 (11)	0.0014 (10)
C2	0.0540 (15)	0.0361 (13)	0.0442 (14)	-0.0040 (11)	0.0134 (12)	0.0013 (11)
C3	0.100 (3)	0.0369 (15)	0.074 (2)	-0.0043 (16)	0.011 (2)	0.0045 (15)
C4	0.143 (4)	0.0470 (19)	0.089 (3)	0.012 (2)	0.000 (3)	0.0246 (19)
C5	0.127 (3)	0.076 (3)	0.063 (2)	0.014 (2)	-0.016 (2)	0.027 (2)
C6	0.082 (2)	0.0601 (18)	0.0405 (15)	-0.0023 (16)	0.0012 (15)	0.0051 (14)
C7	0.0377 (12)	0.0358 (12)	0.0304 (11)	-0.0014 (9)	0.0139 (10)	0.0016 (9)
C8	0.0421 (13)	0.0392 (13)	0.0388 (12)	0.0001 (10)	0.0159 (11)	0.0020 (10)
C9	0.0423 (14)	0.0621 (18)	0.0660 (18)	0.0035 (13)	0.0228 (13)	0.0020 (15)
C10	0.0417 (15)	0.077 (2)	0.081 (2)	-0.0157 (15)	0.0193 (15)	-0.0068 (18)
C11	0.0594 (17)	0.0566 (17)	0.070 (2)	-0.0251 (15)	0.0206 (16)	-0.0159 (15)
C12	0.0536 (15)	0.0398 (13)	0.0512 (15)	-0.0050 (11)	0.0221 (13)	-0.0055 (11)
N1	0.0365 (10)	0.0405 (11)	0.0274 (9)	-0.0006 (8)	0.0124 (8)	-0.0025 (8)
N2	0.0530 (13)	0.0391 (12)	0.0546 (14)	-0.0058 (10)	0.0065 (11)	-0.0084 (11)
O1	0.0866 (14)	0.0501 (10)	0.0439 (10)	-0.0153 (10)	0.0391 (10)	-0.0172 (8)
O2	0.0391 (9)	0.0486 (11)	0.0826 (13)	0.0060 (8)	0.0268 (9)	-0.0022 (10)
O3	0.0725 (13)	0.0634 (13)	0.0454 (11)	0.0022 (11)	0.0095 (10)	0.0042 (10)
O4	0.0606 (14)	0.0777 (16)	0.108 (2)	-0.0295 (12)	-0.0037 (13)	0.0032 (14)
Cl1	0.0585 (4)	0.0417 (3)	0.0616 (4)	0.0036 (3)	0.0253 (3)	-0.0108 (3)
S1	0.0433 (3)	0.0341 (3)	0.0387 (3)	-0.0010 (2)	0.0221 (3)	-0.0057 (2)

Geometric parameters (Å, °)

C1—C6	1.379 (4)	C8—C9	1.383 (3)
C1—C2	1.393 (3)	C8—C11	1.732 (2)
C1—S1	1.774 (2)	C9—C10	1.375 (4)
C2—C3	1.370 (4)	C9—H9	0.9300
C2—N2	1.471 (3)	C10—C11	1.373 (4)
C3—C4	1.370 (5)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.379 (4)
C4—C5	1.375 (5)	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.384 (5)	N1—S1	1.6114 (18)
C5—H5	0.9300	N1—H1N	0.838 (16)
C6—H6	0.9300	N2—O3	1.211 (3)
C7—C12	1.385 (3)	N2—O4	1.216 (3)
C7—C8	1.390 (3)	O1—S1	1.4241 (17)
C7—N1	1.431 (3)	O2—S1	1.4230 (18)
C6—C1—C2	118.5 (2)	C10—C9—C8	119.5 (3)
C6—C1—S1	118.8 (2)	C10—C9—H9	120.2
C2—C1—S1	122.70 (19)	C8—C9—H9	120.2
C3—C2—C1	121.7 (3)	C11—C10—C9	120.3 (3)
C3—C2—N2	116.6 (2)	C11—C10—H10	119.8
C1—C2—N2	121.7 (2)	C9—C10—H10	119.8
C4—C3—C2	119.0 (3)	C10—C11—C12	120.4 (3)
C4—C3—H3	120.5	C10—C11—H11	119.8
C2—C3—H3	120.5	C12—C11—H11	119.8
C3—C4—C5	120.5 (3)	C11—C12—C7	120.1 (2)
C3—C4—H4	119.7	C11—C12—H12	119.9
C5—C4—H4	119.7	C7—C12—H12	119.9
C4—C5—C6	120.4 (3)	C7—N1—S1	122.05 (15)
C4—C5—H5	119.8	C7—N1—H1N	116.9 (17)
C6—C5—H5	119.8	S1—N1—H1N	112.4 (17)
C1—C6—C5	119.9 (3)	O3—N2—O4	125.1 (3)
C1—C6—H6	120.1	O3—N2—C2	118.0 (2)
C5—C6—H6	120.1	O4—N2—C2	116.9 (2)
C12—C7—C8	119.0 (2)	O2—S1—O1	119.96 (12)
C12—C7—N1	119.4 (2)	O2—S1—N1	107.04 (11)
C8—C7—N1	121.6 (2)	O1—S1—N1	106.75 (10)
C9—C8—C7	120.5 (2)	O2—S1—C1	107.77 (11)
C9—C8—C11	119.2 (2)	O1—S1—C1	107.01 (12)
C7—C8—C11	120.26 (18)	N1—S1—C1	107.80 (11)
C6—C1—C2—C3	0.0 (4)	C10—C11—C12—C7	-2.2 (4)
S1—C1—C2—C3	178.9 (2)	C8—C7—C12—C11	0.4 (4)
C6—C1—C2—N2	178.1 (3)	N1—C7—C12—C11	178.0 (2)
S1—C1—C2—N2	-3.0 (4)	C12—C7—N1—S1	76.6 (3)
C1—C2—C3—C4	1.2 (5)	C8—C7—N1—S1	-105.9 (2)

N2—C2—C3—C4	-177.0 (4)	C3—C2—N2—O3	121.6 (3)
C2—C3—C4—C5	-1.0 (7)	C1—C2—N2—O3	-56.6 (3)
C3—C4—C5—C6	-0.4 (8)	C3—C2—N2—O4	-56.8 (4)
C2—C1—C6—C5	-1.4 (5)	C1—C2—N2—O4	125.0 (3)
S1—C1—C6—C5	179.7 (3)	C7—N1—S1—O2	-169.32 (17)
C4—C5—C6—C1	1.6 (7)	C7—N1—S1—O1	-39.7 (2)
C12—C7—C8—C9	2.1 (4)	C7—N1—S1—C1	75.0 (2)
N1—C7—C8—C9	-175.4 (2)	C6—C1—S1—O2	147.7 (2)
C12—C7—C8—C11	-178.01 (18)	C2—C1—S1—O2	-31.2 (2)
N1—C7—C8—C11	4.5 (3)	C6—C1—S1—O1	17.4 (3)
C7—C8—C9—C10	-2.9 (4)	C2—C1—S1—O1	-161.5 (2)
C11—C8—C9—C10	177.2 (2)	C6—C1—S1—N1	-97.1 (2)
C8—C9—C10—C11	1.2 (5)	C2—C1—S1—N1	84.0 (2)
C9—C10—C11—C12	1.3 (5)		

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...O1 ⁱ	0.84 (2)	2.17 (2)	2.844 (2)	138 (2)
N1—H1N...O3	0.84 (2)	2.49 (2)	3.099 (3)	130 (2)

Symmetry code: (i) *x*, -*y*+3/2, *z*+1/2.