

2-Chloro-*N*-(4-nitrobenzoyl)benzenesulfonamide

P. A. Suchetan,^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

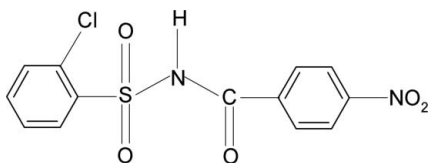
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.033; wR factor = 0.087; data-to-parameter ratio = 11.0.

In the title compound, $\text{C}_{13}\text{H}_9\text{ClN}_2\text{O}_5\text{S}$, the N—H bond is *trans* to the C=O bond. The dihedral angle between the two aromatic rings is $85.4(1)^\circ$. In the crystal, molecules are linked into zigzag $C(4)$ chains along the b axis through N—H...O hydrogen bonds.

Related literature

For our study of the effect of substituents on the structures of *N*-(aryl)-amides, see: Gowda *et al.* (2000), of *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and of *N*-(*p*-substituted-benzoyl)-*p*-substituted-benzenesulfonamides, see: Gowda *et al.* (2010); Suchetan *et al.* (2011).



Experimental

Crystal data

$\text{C}_{13}\text{H}_9\text{ClN}_2\text{O}_5\text{S}$

$M_r = 340.73$

Monoclinic, $P2_1$

$a = 11.097(2)$ Å

$b = 5.3063(7)$ Å

$c = 12.319(2)$ Å

$\beta = 104.24(2)^\circ$

$V = 703.10(19)$ Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 0.45$ mm⁻¹

$T = 293$ K

$0.48 \times 0.16 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

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

Diffraction, 2009)

$T_{\min} = 0.815$, $T_{\max} = 0.949$

2641 measured reflections

2213 independent reflections

2072 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.087$

$S = 1.06$

2213 reflections

202 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.23$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Absolute structure: Flack (1983),

607 Friedel pairs

Flack parameter: 0.05 (8)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O1^i$	0.85 (2)	2.11 (2)	2.941 (3)	168 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; 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: BT5494).

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supporting information

Acta Cryst. (2011). E67, o930 [doi:10.1107/S1600536811009913]

2-Chloro-*N*-(4-nitrobenzoyl)benzenesulfonamide

P. A. Suchetan, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are important constituents of many biologically significant compounds. As a part of studying the effect of substituents on the structures of this class of compounds (Gowda *et al.*, 2000, 2007, 2010; Suchetan *et al.*, 2011), the structure of 2-chloro-*N*-(4-nitrobenzoyl)-benzenesulfonamide (I) has been determined (Fig. 1). The conformation of the N—C bond in the C—SO₂—NH—C(O) segment has *gauche* torsions with respect to the S=O bonds. Further, the N—H bond in the C—SO₂—NH—C(O) segment is *anti* to the C=O bond, similar to those observed in 2-chloro-*N*-(4-methylbenzoyl)-benzenesulfonamide (II) (Gowda *et al.*, 2010) and 2-methyl-*N*-(4-nitrobenzoyl)-benzenesulfonamide (III) (Suchetan *et al.*, 2011).

The molecules are twisted at the S atoms with the C—S(O₂)—NH—C(O) torsional angle of 61.7 (3)°, compared to the values of 60.4 (3)° in (II) and 61.8 (5)° in (III).

The dihedral angle between the sulfonyl benzene ring and the —SO₂—NH—C—O segment is 86.7 (1)°, compared to the values of 89.4 (1)° in (II) and 86.8 (2)° in (III).

The dihedral angle between the sulfonyl and the benzoyl benzene rings is 85.4 (1)°, compared to the values of 89.1 (2)° in (II) and 83.8 (2)° in (III).

The packing of molecules in the crystal linked by of N—H···O hydrogen bonds (Table 1) is shown in Fig. 2.

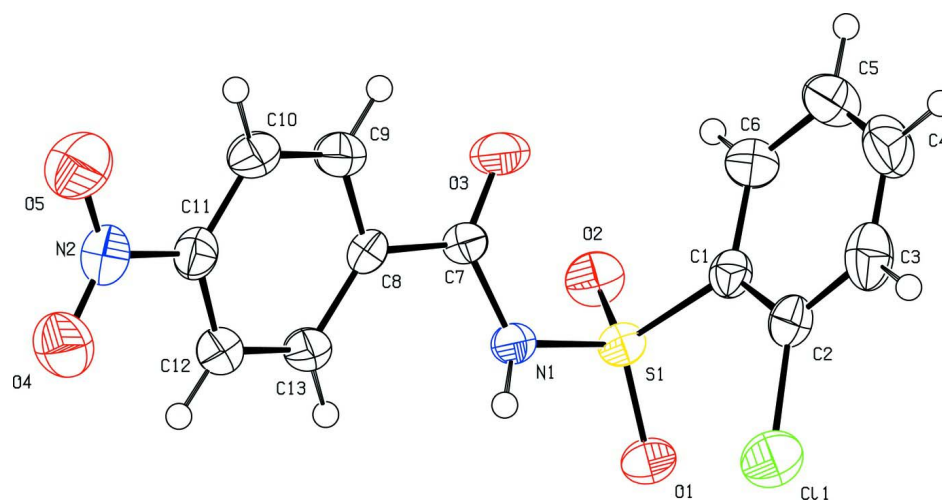
S2. Experimental

The title compound was prepared by refluxing a mixture of 4-nitrobenzoic acid, 2-chlorobenzenesulfonamide and phosphorous oxychloride for 3 hr 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 reprecipitated by acidifying the filtered solution with dilute HCl. It was filtered, dried and recrystallized.

Rod like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its toluene solution at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H distance = 0.93 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

**Figure 1**

Molecular structure of the title compound, showing the atom- labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

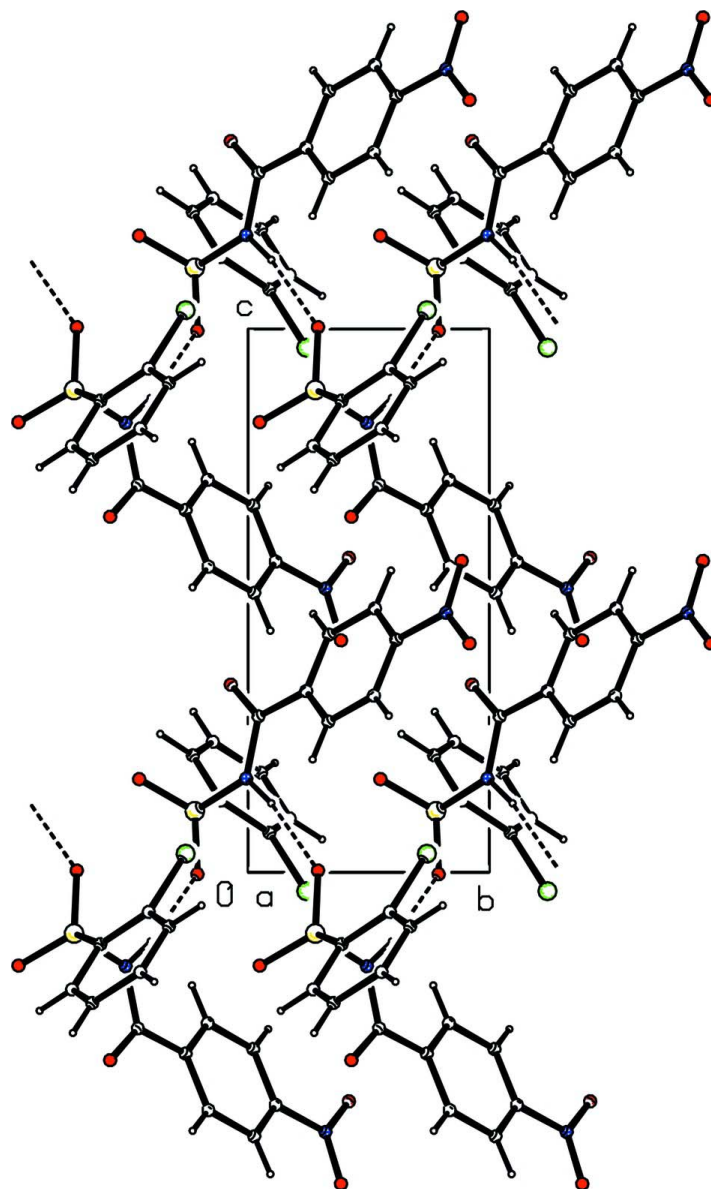


Figure 2

Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

2-Chloro-*N*-(4-nitrobenzoyl)benzenesulfonamide

Crystal data

$C_{13}H_9ClN_2O_5S$

$M_r = 340.73$

Monoclinic, $P2_1$

Hall symbol: $P\ 2y_b$

$a = 11.097\ (2)\ \text{\AA}$

$b = 5.3063\ (7)\ \text{\AA}$

$c = 12.319\ (2)\ \text{\AA}$

$\beta = 104.24\ (2)^\circ$

$V = 703.10\ (19)\ \text{\AA}^3$

$Z = 2$

$F(000) = 348$

$D_x = 1.609\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1433 reflections

$\theta = 2.9\text{--}27.8^\circ$

$\mu = 0.45\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Rod, colourless

$0.48 \times 0.16 \times 0.12\ \text{mm}$

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 and φ
scans
Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.815$, $T_{\max} = 0.949$

2641 measured reflections
2213 independent reflections
2072 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.8^\circ$
 $h = -13 \rightarrow 8$
 $k = -5 \rightarrow 6$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.087$
 $S = 1.06$
2213 reflections
202 parameters
2 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
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.2456P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$
Absolute structure: Flack (1983), 607 Friedel
pairs
Absolute structure parameter: 0.05 (8)

Special details

Experimental. *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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.24217 (8)	0.24133 (17)	-0.03446 (7)	0.0518 (2)
S1	0.40651 (6)	-0.22075 (13)	0.11243 (5)	0.02944 (16)
O1	0.40724 (17)	-0.2097 (5)	-0.00329 (14)	0.0383 (5)
O2	0.4343 (2)	-0.4512 (4)	0.17156 (18)	0.0423 (5)
O3	0.4791 (2)	-0.0713 (5)	0.34556 (16)	0.0495 (6)
O4	0.9366 (2)	0.9181 (5)	0.4214 (2)	0.0607 (7)
O5	0.8756 (3)	0.8856 (6)	0.5731 (2)	0.0675 (8)
N1	0.5096 (2)	-0.0071 (5)	0.17282 (18)	0.0300 (5)
H1N	0.528 (3)	0.097 (5)	0.127 (2)	0.036*
N2	0.8742 (2)	0.8214 (5)	0.4779 (2)	0.0435 (6)
C1	0.2624 (2)	-0.1120 (5)	0.1320 (2)	0.0308 (6)
C2	0.1961 (3)	0.0885 (6)	0.0729 (2)	0.0352 (6)

C3	0.0884 (3)	0.1720 (7)	0.0994 (3)	0.0497 (8)
H3	0.0442	0.3070	0.0608	0.060*
C4	0.0468 (3)	0.0537 (9)	0.1834 (3)	0.0551 (10)
H4	-0.0251	0.1111	0.2013	0.066*
C5	0.1098 (3)	-0.1457 (8)	0.2402 (3)	0.0543 (10)
H5	0.0800	-0.2257	0.2955	0.065*
C6	0.2182 (3)	-0.2287 (8)	0.2153 (2)	0.0423 (7)
H6	0.2616	-0.3635	0.2547	0.051*
C7	0.5325 (2)	0.0433 (6)	0.2870 (2)	0.0323 (6)
C8	0.6273 (2)	0.2411 (6)	0.3335 (2)	0.0319 (6)
C9	0.6257 (3)	0.3356 (7)	0.4375 (2)	0.0416 (8)
H9	0.5698	0.2706	0.4755	0.050*
C10	0.7064 (3)	0.5262 (7)	0.4858 (2)	0.0429 (8)
H10	0.7047	0.5926	0.5552	0.052*
C11	0.7893 (3)	0.6146 (6)	0.4280 (2)	0.0366 (7)
C12	0.7948 (3)	0.5216 (7)	0.3252 (2)	0.0432 (8)
H12	0.8525	0.5835	0.2885	0.052*
C13	0.7121 (3)	0.3329 (7)	0.2776 (2)	0.0399 (8)
H13	0.7137	0.2678	0.2079	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0584 (5)	0.0462 (5)	0.0490 (4)	0.0055 (4)	0.0095 (3)	0.0178 (4)
S1	0.0344 (3)	0.0269 (3)	0.0271 (3)	0.0019 (3)	0.0077 (2)	-0.0002 (3)
O1	0.0459 (11)	0.0410 (12)	0.0292 (9)	0.0005 (11)	0.0119 (8)	-0.0070 (10)
O2	0.0515 (13)	0.0307 (12)	0.0448 (12)	0.0056 (10)	0.0118 (10)	0.0043 (10)
O3	0.0589 (14)	0.0627 (17)	0.0287 (10)	-0.0198 (12)	0.0145 (9)	-0.0008 (11)
O4	0.0569 (15)	0.0591 (17)	0.0662 (16)	-0.0189 (13)	0.0157 (13)	-0.0034 (14)
O5	0.0776 (18)	0.069 (2)	0.0533 (15)	-0.0174 (16)	0.0117 (13)	-0.0253 (14)
N1	0.0318 (12)	0.0338 (14)	0.0246 (11)	-0.0045 (10)	0.0071 (9)	0.0005 (10)
N2	0.0382 (13)	0.0413 (17)	0.0470 (14)	0.0011 (12)	0.0025 (11)	-0.0015 (13)
C1	0.0294 (13)	0.0312 (15)	0.0301 (13)	-0.0006 (12)	0.0040 (10)	-0.0053 (12)
C2	0.0326 (14)	0.0354 (16)	0.0343 (14)	-0.0034 (12)	0.0019 (11)	-0.0037 (13)
C3	0.0350 (16)	0.050 (2)	0.059 (2)	0.0079 (15)	0.0018 (14)	-0.0061 (17)
C4	0.0352 (17)	0.073 (3)	0.059 (2)	0.0004 (18)	0.0166 (15)	-0.016 (2)
C5	0.0476 (19)	0.071 (3)	0.0496 (18)	-0.0089 (18)	0.0217 (15)	-0.0027 (18)
C6	0.0429 (15)	0.0456 (17)	0.0392 (14)	-0.0023 (17)	0.0119 (11)	0.0049 (18)
C7	0.0304 (14)	0.0413 (17)	0.0253 (12)	0.0017 (13)	0.0071 (10)	0.0036 (12)
C8	0.0270 (12)	0.0413 (18)	0.0244 (11)	0.0015 (13)	0.0007 (9)	0.0043 (13)
C9	0.0421 (16)	0.053 (2)	0.0318 (13)	-0.0083 (14)	0.0129 (12)	-0.0023 (14)
C10	0.0453 (18)	0.055 (2)	0.0290 (14)	-0.0057 (16)	0.0106 (12)	-0.0090 (15)
C11	0.0304 (14)	0.0410 (18)	0.0353 (15)	0.0030 (13)	0.0024 (11)	-0.0017 (13)
C12	0.0347 (16)	0.061 (2)	0.0340 (14)	-0.0082 (15)	0.0083 (12)	0.0007 (16)
C13	0.0363 (14)	0.058 (2)	0.0269 (12)	-0.0073 (14)	0.0100 (11)	-0.0067 (14)

Geometric parameters (Å, °)

C11—C2	1.732 (3)	C4—C5	1.363 (5)
S1—O2	1.418 (2)	C4—H4	0.9300
S1—O1	1.4287 (18)	C5—C6	1.384 (4)
S1—N1	1.652 (2)	C5—H5	0.9300
S1—C1	1.771 (3)	C6—H6	0.9300
O3—C7	1.205 (3)	C7—C8	1.497 (4)
O4—N2	1.210 (3)	C8—C9	1.381 (4)
O5—N2	1.219 (3)	C8—C13	1.383 (4)
N1—C7	1.392 (3)	C9—C10	1.385 (4)
N1—H1N	0.849 (18)	C9—H9	0.9300
N2—C11	1.478 (4)	C10—C11	1.376 (4)
C1—C6	1.387 (4)	C10—H10	0.9300
C1—C2	1.393 (4)	C11—C12	1.375 (4)
C2—C3	1.387 (4)	C12—C13	1.387 (4)
C3—C4	1.382 (5)	C12—H12	0.9300
C3—H3	0.9300	C13—H13	0.9300
O2—S1—O1	119.61 (14)	C6—C5—H5	120.1
O2—S1—N1	108.76 (13)	C5—C6—C1	120.5 (3)
O1—S1—N1	104.36 (12)	C5—C6—H6	119.8
O2—S1—C1	107.47 (13)	C1—C6—H6	119.8
O1—S1—C1	110.47 (12)	O3—C7—N1	120.9 (3)
N1—S1—C1	105.25 (13)	O3—C7—C8	121.9 (2)
C7—N1—S1	121.41 (19)	N1—C7—C8	117.2 (2)
C7—N1—H1N	122 (2)	C9—C8—C13	120.0 (3)
S1—N1—H1N	114 (2)	C9—C8—C7	116.2 (2)
O4—N2—O5	124.2 (3)	C13—C8—C7	123.8 (2)
O4—N2—C11	118.2 (3)	C8—C9—C10	120.6 (3)
O5—N2—C11	117.6 (3)	C8—C9—H9	119.7
C6—C1—C2	119.3 (3)	C10—C9—H9	119.7
C6—C1—S1	116.8 (2)	C11—C10—C9	118.1 (3)
C2—C1—S1	123.8 (2)	C11—C10—H10	120.9
C3—C2—C1	119.7 (3)	C9—C10—H10	120.9
C3—C2—C11	117.6 (3)	C12—C11—C10	122.8 (3)
C1—C2—C11	122.7 (2)	C12—C11—N2	118.7 (3)
C4—C3—C2	119.8 (3)	C10—C11—N2	118.5 (3)
C4—C3—H3	120.1	C11—C12—C13	118.2 (3)
C2—C3—H3	120.1	C11—C12—H12	120.9
C5—C4—C3	120.9 (3)	C13—C12—H12	120.9
C5—C4—H4	119.5	C8—C13—C12	120.3 (3)
C3—C4—H4	119.5	C8—C13—H13	119.8
C4—C5—C6	119.8 (3)	C12—C13—H13	119.8
C4—C5—H5	120.1		
O2—S1—N1—C7	-53.2 (3)	S1—N1—C7—O3	1.3 (4)
O1—S1—N1—C7	178.0 (2)	S1—N1—C7—C8	-179.8 (2)

C1—S1—N1—C7	61.7 (2)	O3—C7—C8—C9	-17.6 (4)
O2—S1—C1—C6	13.3 (3)	N1—C7—C8—C9	163.4 (3)
O1—S1—C1—C6	145.4 (2)	O3—C7—C8—C13	163.5 (3)
N1—S1—C1—C6	-102.5 (2)	N1—C7—C8—C13	-15.4 (4)
O2—S1—C1—C2	-169.9 (2)	C13—C8—C9—C10	1.5 (5)
O1—S1—C1—C2	-37.8 (3)	C7—C8—C9—C10	-177.4 (3)
N1—S1—C1—C2	74.3 (3)	C8—C9—C10—C11	-1.1 (5)
C6—C1—C2—C3	1.3 (4)	C9—C10—C11—C12	0.0 (5)
S1—C1—C2—C3	-175.5 (2)	C9—C10—C11—N2	178.9 (3)
C6—C1—C2—C11	-178.5 (2)	O4—N2—C11—C12	7.4 (4)
S1—C1—C2—C11	4.7 (4)	O5—N2—C11—C12	-173.6 (3)
C1—C2—C3—C4	-0.7 (5)	O4—N2—C11—C10	-171.6 (3)
C11—C2—C3—C4	179.1 (3)	O5—N2—C11—C10	7.5 (4)
C2—C3—C4—C5	-0.5 (6)	C10—C11—C12—C13	0.8 (5)
C3—C4—C5—C6	1.3 (6)	N2—C11—C12—C13	-178.1 (3)
C4—C5—C6—C1	-0.7 (5)	C9—C8—C13—C12	-0.7 (5)
C2—C1—C6—C5	-0.5 (4)	C7—C8—C13—C12	178.2 (3)
S1—C1—C6—C5	176.4 (3)	C11—C12—C13—C8	-0.5 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ⁱ	0.85 (2)	2.11 (2)	2.941 (3)	168 (3)

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