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N-Benzoyl-3-nitrobenzenesulfonamide

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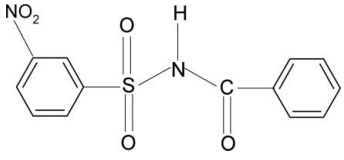
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.093; data-to-parameter ratio = 14.3.

In the title compound, $C_{13}H_{10}N_2O_5S$, the dihedral angle between the phenyl and benzene rings is 86.7 (1)°. In the crystal, molecules are linked into zigzag $C(4)$ chains running along the b axis *via* N—H···O hydrogen bonds.

Related literature

For our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Bowes *et al.* (2003); Gowda *et al.* (2004), on *N*-(aryl)-methanesulfonamides, see: Jayalakshmi & Gowda (2004), on *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2003), on *N*-(substitutedbenzoyl)-arylsulfonamides, see: Suchetan *et al.* (2010) and on *N*-chloroarylamides, see: Gowda *et al.* (1996).



Experimental

Crystal data

$C_{13}H_{10}N_2O_5S$
M_r = 306.29
Monoclinic, $P2_1/c$
a = 11.546 (1) Å
b = 5.0302 (5) Å
c = 23.387 (2) Å
β = 93.69 (1)°
V = 1355.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 K
0.48 × 0.20 × 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} = 0.884, T_{max} = 0.959
4929 measured reflections
2768 independent reflections
2225 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 1.05
2768 reflections
193 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.81 (2)	2.15 (2)	2.954 (2)	172 (2)

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

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: BT5732).

References

Bowes, K. F., Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2003). *Acta Cryst.* C**59**, o1–o3.
Gowda, B. T., Dou, S. Q. & Weiss, A. (1996). *Z. Naturforsch. Teil A*, **51**, 627–636.
Gowda, B. T., Jyothi, K., Kozisek, J. & Fuess, H. (2003). *Z. Naturforsch. Teil A*, **58**, 656–660.
Gowda, B. T., Svoboda, I. & Fuess, H. (2004). *Z. Naturforsch. Teil A*, **59**, 845–852.
Jayalakshmi, K. L. & Gowda, B. T. (2004). *Z. Naturforsch. Teil A*, **59**, 491–500.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010). *Acta Cryst.* E**66**, o1024.

supporting information

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N-Benzoyl-3-nitrobenzenesulfonamide

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

S1. Comment

Diaryl acylsulfonamides are known as potent antitumor agents against a broad spectrum of human tumor xenografts in nude mice. As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Bowes *et al.*, 2003; Gowda *et al.*, 2004), *N*-(aryl)-methanesulfonamides (Jayalakshmi & Gowda, 2004), *N*-(aryl)-aryl-sulfonamides (Gowda *et al.*, 2003); *N*-(substitutedbenzoyl)-arylsulfonamides (Suchetan *et al.*, 2010) and *N*-chloro-aryl-sulfonamides (Gowda *et al.*, 1996), in the present work, the crystal structure of *N*-(benzoyl)- 3-nitrobenzenesulfonamide (I) has been determined (Fig.1).

The conformations of the N—H and C=O bonds in the C—SO_2—NH—C(O) segment are *anti* to each other (Fig.1), similar to that observed in *N*-(benzoyl)-2-methylbenzenesulfonamide (II)(Suchetan *et al.*, 2010). Further, The N—C bond in the C—SO_2—NH—C segment has *gauche* torsion with respect to the S═O bonds. In (I), the conformation between the N—H bond and the *meta*-nitro group in the sulfonyl benzene ring is *syn*.

The molecule is twisted at the *S* atom with the torsional angle of -62.80 (17)°, compared to the value of 68.8 (4)° in (II).

The dihedral angle between the sulfonyl benzene ring and the —SO_2—NH—C—O segment is 79.2 (1)°, compared to the value of 84.8 (1)° in (II). Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene rings is 86.7 (1)°, compared to the value of 73.9 (1)° in (II).

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

S2. Experimental

The title compound was prepared by refluxing a mixture of benzoic acid (0.02 mole), 3-nitrobenzenesulfonamide (0.02 mole) and excess phosphorous oxy chloride for 3 h on a water bath. The resultant mixture was cooled and poured into crushed ice. The solid, *N*-(benzoyl)-3-nitrobenzenesulfonamide, 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 C—H = 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.

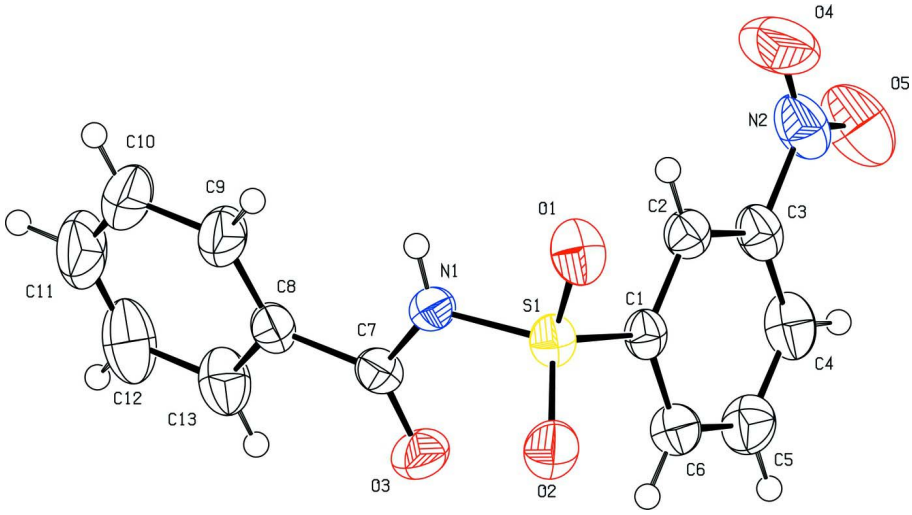

Figure 1

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

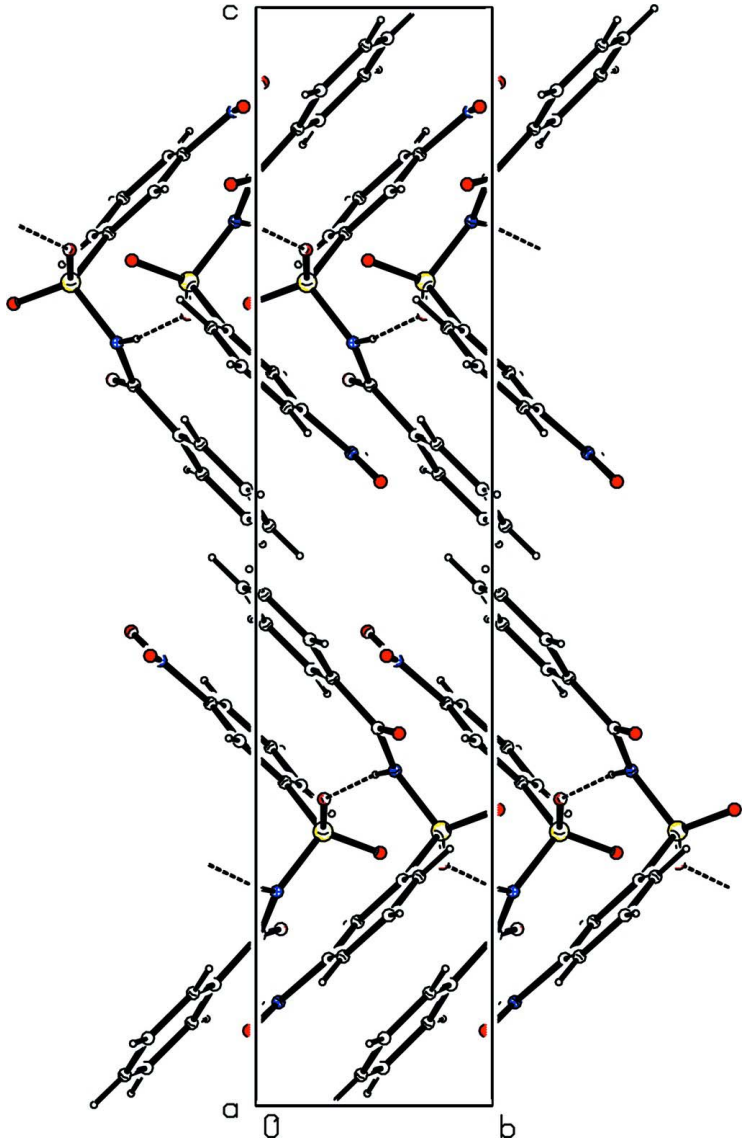

Figure 2

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

N-Benzoyl-3-nitrobenzenesulfonamide

Crystal data

$C_{13}H_{10}N_2O_5S$
$M_r = 306.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.546\ (1)$ Å
$b = 5.0302\ (5)$ Å
$c = 23.387\ (2)$ Å
$\beta = 93.69\ (1)°$
$V = 1355.5\ (2)$ Å^3
$Z = 4$

$F(000) = 632$
$D_x = 1.501$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1881 reflections
$\theta = 2.6–27.8°$
$\mu = 0.26$ mm^{-1}
$T = 293$ K
Rod, colourless
0.48 × 0.20 × 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 and phi scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.884, T_{max} = 0.959
4929 measured reflections
2768 independent reflections
2225 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 26.4°, θ_{min} = 2.6°
h = −9→14
k = −6→3
l = −24→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 1.05
2768 reflections
193 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
$w = 1/[\sigma^2(F_o^2) + (0.0313P)^2 + 0.8565P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.23224 (16)	0.3781 (4)	0.79448 (8)	0.0329 (4)
C2	0.27622 (17)	0.5700 (4)	0.83229 (8)	0.0363 (4)
H2	0.3546	0.6143	0.8346	0.044*
C3	0.19897 (18)	0.6929 (4)	0.86639 (8)	0.0405 (5)
C4	0.08278 (19)	0.6308 (5)	0.86402 (9)	0.0496 (6)
H4	0.0329	0.7184	0.8874	0.059*
C5	0.04152 (19)	0.4376 (5)	0.82661 (10)	0.0514 (6)
H5	−0.0367	0.3923	0.8249	0.062*
C6	0.11592 (18)	0.3100 (4)	0.79148 (9)	0.0431 (5)
H6	0.0879	0.1795	0.7661	0.052*
C7	0.25229 (17)	0.4841 (4)	0.65601 (8)	0.0357 (4)
C8	0.27975 (17)	0.6755 (4)	0.61050 (8)	0.0374 (4)
C9	0.3905 (2)	0.7661 (6)	0.60253 (10)	0.0608 (7)

H9	0.4533	0.6984	0.6248	0.073*
C10	0.4082 (3)	0.9574 (7)	0.56151 (11)	0.0812 (10)
H10	0.4828	1.0197	0.5567	0.097*
C11	0.3168 (3)	1.0554 (6)	0.52804 (11)	0.0782 (9)
H11	0.3290	1.1853	0.5008	0.094*
C12	0.2079 (3)	0.9630 (6)	0.53456 (11)	0.0722 (8)
H12	0.1460	1.0284	0.5113	0.087*
C13	0.1884 (2)	0.7726 (5)	0.57538 (9)	0.0557 (6)
H13	0.1137	0.7094	0.5793	0.067*
N1	0.34339 (14)	0.4120 (3)	0.69483 (7)	0.0339 (4)
H1N	0.4037 (15)	0.495 (4)	0.6984 (9)	0.041*
N2	0.2428 (2)	0.9045 (4)	0.90566 (8)	0.0564 (5)
O1	0.43954 (12)	0.2116 (3)	0.77920 (6)	0.0501 (4)
O2	0.27356 (14)	−0.0250 (3)	0.73033 (6)	0.0518 (4)
O3	0.15637 (12)	0.3948 (3)	0.66098 (7)	0.0551 (4)
O4	0.34651 (19)	0.9464 (4)	0.90974 (9)	0.0856 (7)
O5	0.17200 (19)	1.0273 (4)	0.93168 (8)	0.0857 (7)
S1	0.32724 (4)	0.21577 (10)	0.74967 (2)	0.03588 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0396 (10)	0.0295 (9)	0.0295 (9)	0.0041 (8)	0.0026 (8)	0.0043 (8)
C2	0.0412 (11)	0.0323 (10)	0.0353 (10)	0.0011 (8)	0.0017 (8)	0.0028 (8)
C3	0.0548 (12)	0.0338 (11)	0.0325 (10)	0.0074 (9)	0.0005 (9)	0.0014 (9)
C4	0.0496 (13)	0.0580 (14)	0.0417 (12)	0.0193 (11)	0.0074 (10)	0.0023 (11)
C5	0.0377 (11)	0.0672 (16)	0.0496 (13)	0.0024 (11)	0.0051 (10)	0.0056 (12)
C6	0.0444 (11)	0.0441 (12)	0.0404 (11)	−0.0023 (10)	0.0006 (9)	0.0018 (10)
C7	0.0380 (10)	0.0349 (10)	0.0337 (10)	−0.0010 (9)	−0.0007 (8)	−0.0030 (8)
C8	0.0452 (11)	0.0374 (11)	0.0295 (9)	0.0010 (9)	0.0019 (8)	−0.0013 (8)
C9	0.0549 (14)	0.0849 (19)	0.0418 (12)	−0.0150 (13)	−0.0014 (10)	0.0196 (13)
C10	0.088 (2)	0.103 (2)	0.0523 (15)	−0.0375 (19)	0.0038 (14)	0.0266 (17)
C11	0.122 (3)	0.0672 (19)	0.0459 (14)	−0.0052 (18)	0.0111 (16)	0.0204 (14)
C12	0.091 (2)	0.079 (2)	0.0462 (14)	0.0293 (17)	0.0040 (14)	0.0187 (14)
C13	0.0570 (14)	0.0676 (16)	0.0423 (12)	0.0123 (12)	0.0013 (10)	0.0089 (12)
N1	0.0338 (8)	0.0331 (9)	0.0349 (8)	−0.0041 (7)	0.0012 (7)	0.0023 (7)
N2	0.0812 (15)	0.0457 (11)	0.0419 (11)	0.0100 (11)	0.0005 (10)	−0.0078 (9)
O1	0.0453 (8)	0.0582 (10)	0.0465 (8)	0.0199 (7)	−0.0001 (7)	0.0066 (8)
O2	0.0786 (11)	0.0269 (8)	0.0513 (9)	−0.0036 (7)	0.0143 (8)	−0.0034 (7)
O3	0.0397 (8)	0.0684 (11)	0.0561 (9)	−0.0162 (8)	−0.0053 (7)	0.0129 (9)
O4	0.0849 (15)	0.0825 (15)	0.0889 (15)	−0.0178 (12)	0.0012 (12)	−0.0401 (12)
O5	0.1106 (16)	0.0775 (14)	0.0689 (12)	0.0278 (12)	0.0056 (11)	−0.0327 (11)
S1	0.0445 (3)	0.0285 (2)	0.0350 (3)	0.0064 (2)	0.0045 (2)	0.0023 (2)

Geometric parameters (Å, º)

C1—C2	1.383 (3)	C8—C13	1.383 (3)
C1—C6	1.383 (3)	C9—C10	1.383 (3)

C1—S1	1.7654 (19)	C9—H9	0.9300
C2—C3	1.380 (3)	C10—C11	1.365 (4)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.375 (3)	C11—C12	1.357 (4)
C3—N2	1.474 (3)	C11—H11	0.9300
C4—C5	1.372 (3)	C12—C13	1.381 (4)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.384 (3)	C13—H13	0.9300
C5—H5	0.9300	N1—S1	1.6387 (17)
C6—H6	0.9300	N1—H1N	0.810 (15)
C7—O3	1.208 (2)	N2—O4	1.213 (3)
C7—N1	1.392 (2)	N2—O5	1.219 (3)
C7—C8	1.485 (3)	O1—S1	1.4294 (15)
C8—C9	1.382 (3)	O2—S1	1.4209 (15)

C2—C1—C6	121.36 (18)	C10—C9—H9	119.9
C2—C1—S1	119.06 (15)	C11—C10—C9	120.4 (3)
C6—C1—S1	119.58 (15)	C11—C10—H10	119.8
C3—C2—C1	117.24 (19)	C9—C10—H10	119.8
C3—C2—H2	121.4	C12—C11—C10	120.0 (3)
C1—C2—H2	121.4	C12—C11—H11	120.0
C4—C3—C2	122.7 (2)	C10—C11—H11	120.0
C4—C3—N2	119.0 (2)	C11—C12—C13	120.6 (3)
C2—C3—N2	118.3 (2)	C11—C12—H12	119.7
C5—C4—C3	119.0 (2)	C13—C12—H12	119.7
C5—C4—H4	120.5	C12—C13—C8	120.2 (2)
C3—C4—H4	120.5	C12—C13—H13	119.9
C4—C5—C6	120.2 (2)	C8—C13—H13	119.9
C4—C5—H5	119.9	C7—N1—S1	123.21 (13)
C6—C5—H5	119.9	C7—N1—H1N	122.8 (16)
C1—C6—C5	119.5 (2)	S1—N1—H1N	111.6 (15)
C1—C6—H6	120.2	O4—N2—O5	124.3 (2)
C5—C6—H6	120.2	O4—N2—C3	118.2 (2)
O3—C7—N1	119.97 (18)	O5—N2—C3	117.5 (2)
O3—C7—C8	123.34 (18)	O2—S1—O1	120.32 (10)
N1—C7—C8	116.68 (17)	O2—S1—N1	109.49 (9)
C9—C8—C13	118.7 (2)	O1—S1—N1	103.98 (9)
C9—C8—C7	123.57 (19)	O2—S1—C1	107.95 (10)
C13—C8—C7	117.71 (19)	O1—S1—C1	107.39 (9)
C8—C9—C10	120.1 (2)	N1—S1—C1	107.00 (9)
C8—C9—H9	119.9		

C6—C1—C2—C3	−0.8 (3)	C11—C12—C13—C8	0.6 (4)
S1—C1—C2—C3	179.74 (14)	C9—C8—C13—C12	−2.2 (4)
C1—C2—C3—C4	0.3 (3)	C7—C8—C13—C12	176.1 (2)
C1—C2—C3—N2	−177.96 (17)	O3—C7—N1—S1	−2.2 (3)
C2—C3—C4—C5	0.5 (3)	C8—C7—N1—S1	176.65 (14)
N2—C3—C4—C5	178.7 (2)	C4—C3—N2—O4	175.8 (2)

C3—C4—C5—C6	−0.7 (3)	C2—C3—N2—O4	−5.9 (3)
C2—C1—C6—C5	0.6 (3)	C4—C3—N2—O5	−4.7 (3)
S1—C1—C6—C5	−179.95 (16)	C2—C3—N2—O5	173.6 (2)
C4—C5—C6—C1	0.2 (3)	C7—N1—S1—O2	53.95 (18)
O3—C7—C8—C9	−176.0 (2)	C7—N1—S1—O1	−176.27 (16)
N1—C7—C8—C9	5.2 (3)	C7—N1—S1—C1	−62.80 (17)
O3—C7—C8—C13	5.8 (3)	C2—C1—S1—O2	160.32 (15)
N1—C7—C8—C13	−173.01 (19)	C6—C1—S1—O2	−19.17 (18)
C13—C8—C9—C10	2.3 (4)	C2—C1—S1—O1	29.21 (18)
C7—C8—C9—C10	−175.9 (2)	C6—C1—S1—O1	−150.28 (16)
C8—C9—C10—C11	−1.0 (5)	C2—C1—S1—N1	−81.92 (16)
C9—C10—C11—C12	−0.7 (5)	C6—C1—S1—N1	98.59 (17)
C10—C11—C12—C13	0.8 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.81 (2)	2.15 (2)	2.954 (2)	172 (2)

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