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N-(4-Methylbenzoyl)-4-nitrobenzenesulfonamide

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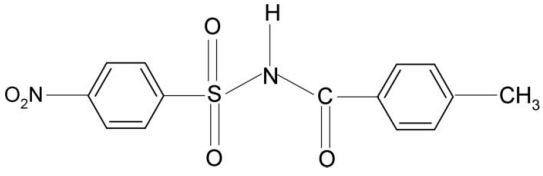
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.062; wR factor = 0.152; data-to-parameter ratio = 14.1.

In the title compound, $C_{14}H_{12}N_2O_5S$, the dihedral angle between the nitrophenyl group and the –S—NH—C—O fragment is 80.74 (17)° and that between the nitrophenyl and methylphenyl groups is 87.66 (14)°. The C—S—N—C torsion angle at the S—N bond is −67.0 (3)°. In the crystal, molecules are linked into $C(4)$ chains *via* N—H···O hydrogen bonds.

Related literature

For our studies on the effects of substituents on the structures and other aspects of *N*-arylamides, see: Gowda *et al.* (1999, 2006). For *N*-aryl-methanesulfonamides, see: Gowda *et al.* (2007). For *N*-(substituted-benzoyl)-arylsulfonamides, see: Suchetan *et al.* (2010). For *N*-chloroarylamides, see: Jyothi & Gowda (2004). For *N*-bromoarylsulfonamides, see: Usha & Gowda (2006).



Experimental

Crystal data

$C_{14}H_{12}N_2O_5S$
M_r = 320.32
Orthorhombic, *Pbca*
a = 13.969 (1) Å
b = 9.6591 (6) Å
c = 21.026 (2) Å
V = 2837.0 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 K
0.40 × 0.18 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.905, T_{max} = 0.956
7163 measured reflections
2864 independent reflections
1835 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.152
S = 1.16
2863 reflections
203 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O3^{i}	0.85 (2)	2.16 (2)	2.994 (4)	168 (4)

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

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: YK2044).

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supplementary materials

Acta Cryst. (2012). E**68**, o888 [doi:10.1107/S1600536812007854]

N-(4-Methylbenzoyl)-4-nitrobenzenesulfonamide

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

Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-arylamides (Gowda *et al.*, 1999, 2006), *N*-aryl-methanesulfonamides (Gowda *et al.*, 2007), *N*-(substituted-benzoyl)-arylsulfonamides (Suchetan *et al.*, 2010), *N*-chloroarylsulfonamides (Jyothi & Gowda, 2004) and *N*-bromoarylsulfonamides (Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(4-methylbenzoyl)-4-nitrobenzenesulfonamide has been determined (Fig.1).

The conformation of the N—H bond in the C—SO_2—NH—C(O) segment is *anti* with respect to the C=O bond (Fig.1), similar to that observed in *N*-(4-methylbenzoyl)-4-chlorobenzenesulfonamide (I) (Suchetan *et al.*, 2010).

In the title compound, the molecules are twisted at the *S*–*N* bonds with the torsional angle of -67.0 (3)°, compared to the value of 69.0 (2)° in (I).

The dihedral angle between the sulfonyl benzene ring and the —SO_2—NH—C—O segment is 79.6 (1)°, compared to the value of 77.2 (1)° in (I).

The dihedral angle between the sulfonyl and the benzoyl benzene rings is 89.3 (1)°, compared to the value of 89.5 (1)° in (I).

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

Experimental

The title compound was prepared by refluxing a mixture of 4-methylbenzoic acid, 4-nitrobenzenesulfonamide and phosphorous oxy chloride 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 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 from its toluene solution at room temperature.

Refinement

The H atom of the NH group was located in a difference map and restrained to N—H = 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H distances of 0.93Å (*C*-aromatic) and 0.96Å (*C*-methyl).

All H atoms were refined with isotropic displacement parameters were set at 1.2 U_{eq}(C-aromatic, N) and 1.5 U_{eq}(*C*-methyl).

The U^{ij} components of O4 were restrained to approximate isotropic behaviour.

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); 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* (Sheldrick, 2008).

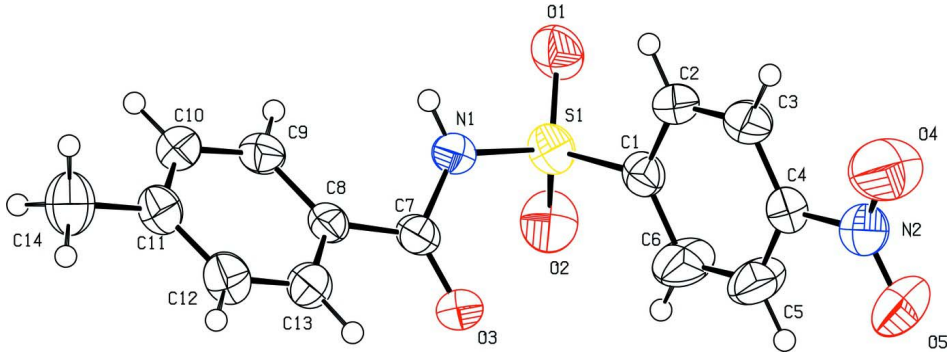

Figure 1

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

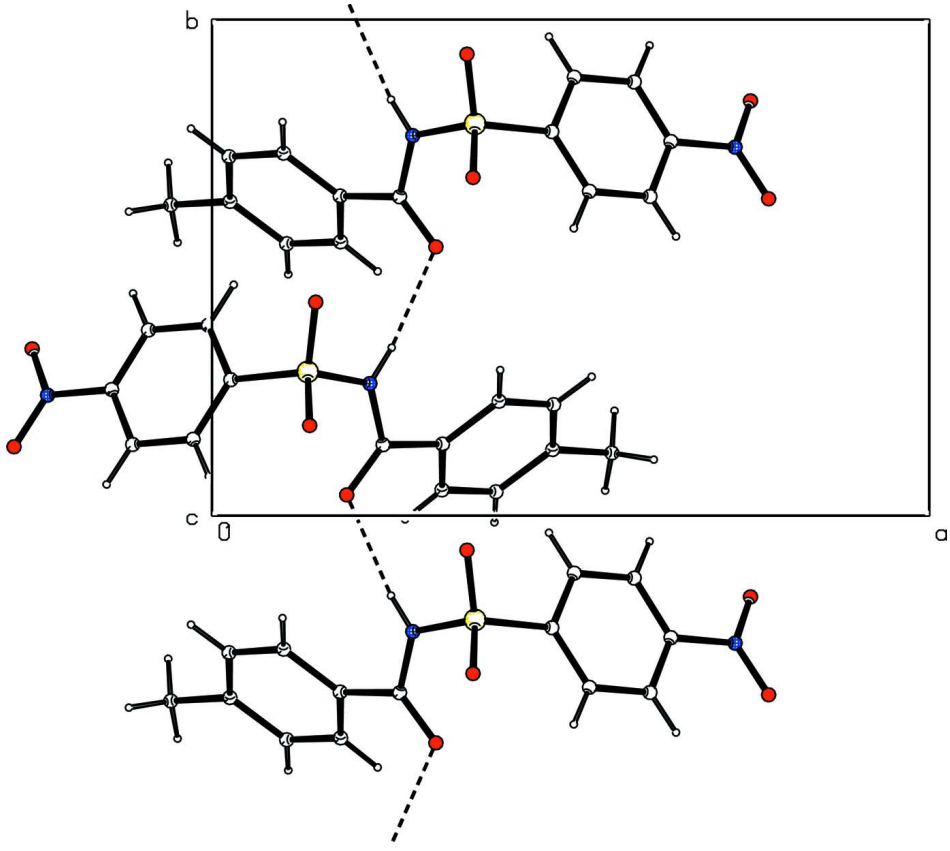

Figure 2

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

N-(4-Methylbenzoyl)-4-nitrobenzenesulfonamide

Crystal data

$C_{14}H_{12}N_2O_5S$
$M_r = 320.32$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.969$ (1) Å
$b = 9.6591$ (6) Å
$c = 21.026$ (2) Å
$V = 2837.0$ (4) Å^3
$Z = 8$
$F(000) = 1328$
$D_x = 1.500$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 818 reflections
$\theta = 2.6$–$27.9°$
$\mu = 0.25$ mm^{-1}
$T = 293$ K
Rod, colourless
0.40 × 0.18 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{min} = 0.905$, $T_{max} = 0.956$
7163 measured reflections
2864 independent reflections
1836 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.9°$
$h = -7 \rightarrow 17$
$k = -7 \rightarrow 12$
$l = -26 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.152$
$S = 1.16$
2863 reflections
203 parameters
7 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
$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 3.5546P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.015$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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 $F^2 > \sigma(F^2)$ 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	0.5261 (3)	0.2260 (4)	0.55537 (17)	0.0407 (9)
C2	0.4949 (3)	0.1161 (4)	0.5903 (2)	0.0490 (10)
H2	0.5302	0.0345	0.5911	0.059*
C3	0.4113 (3)	0.1260 (4)	0.6240 (2)	0.0533 (11)

H3	0.3899	0.0521	0.6486	0.064*
C4	0.3601 (3)	0.2459 (4)	0.62105 (19)	0.0450 (9)
C5	0.3893 (3)	0.3565 (5)	0.5862 (2)	0.0669 (13)
H5	0.3531	0.4372	0.5847	0.080*
C6	0.4737 (4)	0.3461 (5)	0.5534 (2)	0.0708 (14)
H6	0.4956	0.4209	0.5296	0.085*
C7	0.7385 (3)	0.3573 (4)	0.59457 (17)	0.0405 (9)
C8	0.8213 (3)	0.3558 (4)	0.63877 (17)	0.0385 (8)
C9	0.9003 (3)	0.2694 (4)	0.63104 (19)	0.0449 (9)
H9	0.9018	0.2066	0.5975	0.054*
C10	0.9763 (3)	0.2765 (4)	0.67281 (19)	0.0490 (10)
H10	1.0293	0.2199	0.6664	0.059*
C11	0.9750 (3)	0.3668 (4)	0.72441 (19)	0.0463 (10)
C12	0.8959 (3)	0.4520 (4)	0.73124 (19)	0.0497 (10)
H12	0.8938	0.5135	0.7652	0.060*
C13	0.8207 (3)	0.4481 (4)	0.68931 (18)	0.0458 (10)
H13	0.7691	0.5076	0.6948	0.055*
C14	1.0575 (3)	0.3736 (5)	0.7701 (2)	0.0638 (12)
H14A	1.1160	0.3865	0.7469	0.077*
H14B	1.0609	0.2889	0.7939	0.077*
H14C	1.0484	0.4499	0.7988	0.077*
N1	0.7201 (2)	0.2339 (3)	0.56360 (15)	0.0421 (8)
H1N	0.750 (3)	0.161 (3)	0.5748 (17)	0.051*
N2	0.2706 (3)	0.2572 (4)	0.65756 (17)	0.0569 (9)
O1	0.6446 (2)	0.0697 (3)	0.49280 (14)	0.0605 (8)
O2	0.6361 (2)	0.3183 (3)	0.46528 (13)	0.0647 (9)
O3	0.68781 (18)	0.4583 (3)	0.58609 (13)	0.0519 (7)
O4	0.2492 (3)	0.1639 (4)	0.6926 (2)	0.1066 (14)
O5	0.2239 (2)	0.3613 (4)	0.65262 (17)	0.0805 (10)
S1	0.63333 (7)	0.21032 (11)	0.51121 (5)	0.0474 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.042 (2)	0.042 (2)	0.038 (2)	−0.0042 (18)	−0.0084 (17)	0.0030 (17)
C2	0.046 (2)	0.035 (2)	0.067 (3)	0.0027 (18)	−0.002 (2)	0.005 (2)
C3	0.050 (2)	0.039 (2)	0.070 (3)	−0.003 (2)	0.003 (2)	0.012 (2)
C4	0.0384 (19)	0.044 (2)	0.053 (2)	−0.0040 (18)	−0.0034 (19)	0.0001 (18)
C5	0.061 (3)	0.052 (3)	0.088 (4)	0.018 (2)	0.014 (3)	0.024 (3)
C6	0.073 (3)	0.053 (3)	0.087 (3)	0.012 (2)	0.019 (3)	0.039 (3)
C7	0.041 (2)	0.0330 (19)	0.048 (2)	−0.0044 (17)	0.0068 (18)	−0.0038 (18)
C8	0.0351 (18)	0.0321 (18)	0.048 (2)	−0.0054 (16)	0.0051 (17)	−0.0010 (17)
C9	0.046 (2)	0.035 (2)	0.054 (2)	−0.0011 (18)	0.0016 (19)	−0.0090 (18)
C10	0.041 (2)	0.042 (2)	0.064 (3)	0.0021 (18)	0.002 (2)	−0.001 (2)
C11	0.043 (2)	0.049 (2)	0.047 (2)	−0.007 (2)	0.0037 (18)	0.001 (2)
C12	0.048 (2)	0.056 (2)	0.045 (2)	−0.007 (2)	0.0065 (19)	−0.013 (2)
C13	0.040 (2)	0.044 (2)	0.053 (2)	0.0000 (18)	0.0099 (19)	−0.0075 (19)
C14	0.055 (3)	0.085 (3)	0.052 (3)	−0.005 (2)	−0.007 (2)	−0.002 (2)
N1	0.0432 (18)	0.0345 (17)	0.0487 (19)	−0.0012 (14)	−0.0045 (15)	−0.0009 (15)
N2	0.051 (2)	0.056 (2)	0.064 (2)	−0.0004 (19)	0.0042 (19)	0.0004 (19)

O1	0.0604 (18)	0.0604 (18)	0.0606 (18)	−0.0056 (15)	0.0001 (15)	−0.0237 (15)
O2	0.069 (2)	0.081 (2)	0.0444 (16)	−0.0037 (17)	0.0040 (15)	0.0182 (16)
O3	0.0455 (16)	0.0353 (14)	0.0749 (19)	0.0039 (13)	−0.0010 (14)	−0.0027 (14)
O4	0.098 (3)	0.081 (2)	0.140 (3)	0.004 (2)	0.058 (2)	0.026 (2)
O5	0.062 (2)	0.086 (2)	0.094 (3)	0.0287 (19)	0.0031 (18)	0.004 (2)
S1	0.0490 (6)	0.0523 (6)	0.0409 (5)	−0.0053 (5)	−0.0008 (5)	−0.0027 (5)

Geometric parameters (Å, º)

C1—C2	1.362 (5)	C9—H9	0.9300
C1—C6	1.373 (5)	C10—C11	1.392 (5)
C1—S1	1.769 (4)	C10—H10	0.9300
C2—C3	1.369 (5)	C11—C12	1.385 (5)
C2—H2	0.9300	C11—C14	1.502 (5)
C3—C4	1.363 (5)	C12—C13	1.372 (5)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.359 (6)	C13—H13	0.9300
C4—N2	1.471 (5)	C14—H14A	0.9600
C5—C6	1.369 (6)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—H6	0.9300	N1—S1	1.654 (3)
C7—O3	1.218 (4)	N1—H1N	0.852 (19)
C7—N1	1.383 (4)	N2—O4	1.202 (5)
C7—C8	1.484 (5)	N2—O5	1.203 (4)
C8—C13	1.387 (5)	O1—S1	1.421 (3)
C8—C9	1.393 (5)	O2—S1	1.422 (3)
C9—C10	1.380 (5)		
C2—C1—C6	120.3 (4)	C11—C10—H10	119.4
C2—C1—S1	119.1 (3)	C12—C11—C10	117.6 (4)
C6—C1—S1	120.5 (3)	C12—C11—C14	121.3 (4)
C1—C2—C3	119.9 (4)	C10—C11—C14	121.1 (4)
C1—C2—H2	120.1	C13—C12—C11	121.9 (4)
C3—C2—H2	120.1	C13—C12—H12	119.1
C4—C3—C2	118.9 (4)	C11—C12—H12	119.1
C4—C3—H3	120.6	C12—C13—C8	120.4 (4)
C2—C3—H3	120.6	C12—C13—H13	119.8
C5—C4—C3	122.4 (4)	C8—C13—H13	119.8
C5—C4—N2	118.6 (4)	C11—C14—H14A	109.5
C3—C4—N2	119.0 (4)	C11—C14—H14B	109.5
C4—C5—C6	118.2 (4)	H14A—C14—H14B	109.5
C4—C5—H5	120.9	C11—C14—H14C	109.5
C6—C5—H5	120.9	H14A—C14—H14C	109.5
C5—C6—C1	120.4 (4)	H14B—C14—H14C	109.5
C5—C6—H6	119.8	C7—N1—S1	124.7 (3)
C1—C6—H6	119.8	C7—N1—H1N	119 (3)
O3—C7—N1	120.9 (3)	S1—N1—H1N	116 (3)
O3—C7—C8	123.5 (3)	O4—N2—O5	123.0 (4)
N1—C7—C8	115.6 (3)	O4—N2—C4	118.4 (4)
C13—C8—C9	118.6 (4)	O5—N2—C4	118.6 (4)

C13—C8—C7	118.0 (3)	O1—S1—O2	120.89 (19)
C9—C8—C7	123.4 (3)	O1—S1—N1	103.42 (17)
C10—C9—C8	120.4 (4)	O2—S1—N1	109.36 (17)
C10—C9—H9	119.8	O1—S1—C1	108.56 (18)
C8—C9—H9	119.8	O2—S1—C1	108.45 (18)
C9—C10—C11	121.2 (4)	N1—S1—C1	105.02 (16)
C9—C10—H10	119.4		

C6—C1—C2—C3	−0.7 (6)	C14—C11—C12—C13	179.1 (4)
S1—C1—C2—C3	−179.1 (3)	C11—C12—C13—C8	1.0 (6)
C1—C2—C3—C4	1.1 (6)	C9—C8—C13—C12	−1.0 (5)
C2—C3—C4—C5	−0.6 (7)	C7—C8—C13—C12	−179.4 (3)
C2—C3—C4—N2	−179.6 (4)	O3—C7—N1—S1	2.5 (5)
C3—C4—C5—C6	−0.3 (7)	C8—C7—N1—S1	−178.8 (3)
N2—C4—C5—C6	178.7 (4)	C5—C4—N2—O4	−173.7 (5)
C4—C5—C6—C1	0.7 (8)	C3—C4—N2—O4	5.4 (6)
C2—C1—C6—C5	−0.2 (7)	C5—C4—N2—O5	3.9 (6)
S1—C1—C6—C5	178.2 (4)	C3—C4—N2—O5	−177.0 (4)
O3—C7—C8—C13	25.4 (5)	C7—N1—S1—O1	179.3 (3)
N1—C7—C8—C13	−153.2 (3)	C7—N1—S1—O2	49.2 (4)
O3—C7—C8—C9	−152.8 (4)	C7—N1—S1—C1	−67.0 (3)
N1—C7—C8—C9	28.5 (5)	C2—C1—S1—O1	29.9 (4)
C13—C8—C9—C10	−0.3 (6)	C6—C1—S1—O1	−148.6 (4)
C7—C8—C9—C10	177.9 (3)	C2—C1—S1—O2	162.9 (3)
C8—C9—C10—C11	1.8 (6)	C6—C1—S1—O2	−15.5 (4)
C9—C10—C11—C12	−1.8 (6)	C2—C1—S1—N1	−80.2 (3)
C9—C10—C11—C14	179.6 (4)	C6—C1—S1—N1	101.3 (4)
C10—C11—C12—C13	0.4 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.85 (2)	2.16 (2)	2.994 (4)	168 (4)

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