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N-(4-Methylphenyl)-2-nitrobenzenesulfonamide

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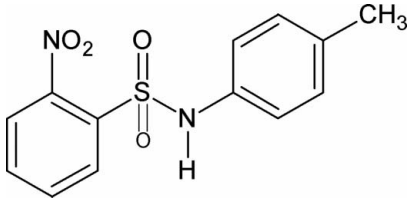
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 15.0.

In the crystal of the title compound, $C_{13}H_{12}N_2O_4S$, the conformation of the N—H bond in the –SO_2—NH– segment is *syn* to the *ortho*-nitro group in the sulfonyl benzene ring. The molecule is twisted at the S—N bond with a torsion angle of 76.55 (18)°. The dihedral angle between the planes of the rings is 72.64 (8)°. In the crystal, molecules are linked by pairs of N—H···O(S) hydrogen bonds to form 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 & Weiss (1994); Saeed *et al.* (2010); Shahwar *et al.* (2012). For *N*-arylsulfonamides, see: Chaithanya *et al.* (2012); Gowda *et al.* (2005). For *N*-chloroarylsulfonamides, see: Gowda & Shetty (2004); Shetty & Gowda (2004).



Experimental

Crystal data

$C_{13}H_{12}N_2O_4S$
M_r = 292.32
Monoclinic, $P2_1/c$
a = 8.2787 (5) Å
b = 11.2017 (7) Å
c = 14.6435 (8) Å
β = 91.116 (5)°
V = 1357.72 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 295 K
0.48 × 0.40 × 0.40 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.888, T_{max} = 0.906
5351 measured reflections
2766 independent reflections
2353 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.06
2766 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.84 (2)	2.27 (2)	3.099 (2)	169 (2)

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

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: RK2377).

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supporting information

Acta Cryst. (2012). E**68**, o2745 [doi:10.1107/S1600536812035866]

N-(4-Methylphenyl)-2-nitrobenzenesulfonamide

U. Chaithanya, Sabine Foro and B. Thimme Gowda

S1. Comment

As a 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 & Weiss, 1994; Saeed *et al.*, 2010; Shahwar *et al.*, 2012); *N*-arylsulfonamides (Chaithanya *et al.*, 2012; Gowda *et al.*, 2005) and *N*-chloroarylsulfonamides (Gowda & Shetty, 2004; Shetty & Gowda, 2004), in the present work, the molecular structure of *N*-(4-methylphenyl)-2-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N–H bond in the –SO_2–NH– segment is *syn* to the *ortho*-nitro group in the sulfonyl benzene ring, similar to that observed in *N*-(4-chlorophenyl)-2-nitrobenzenesulfonamide **I** (Chaithanya *et al.*, 2012). The molecule is twisted at the S–N bond with the torsional angle of 76.55 (18)°, compared to the value of 79.17 (18)° in **I**.

The dihedral angle between the sulfonyl and the anilino rings is 72.64 (8)°, compared to the value of 70.27 (8)° in **I**.

In the crystal structure, 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-methylaniline 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-methylphenyl)-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 (385 K) 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 the aromatic C–H = 0.93Å, methyl C–H = 0.96Å. The amino H atom was freely refined with the N–H distance restrained to 0.86 (2)Å. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq}(C-aromatic, N) and 1.5 U_{eq}(*C*-methyl) of the parent atom.

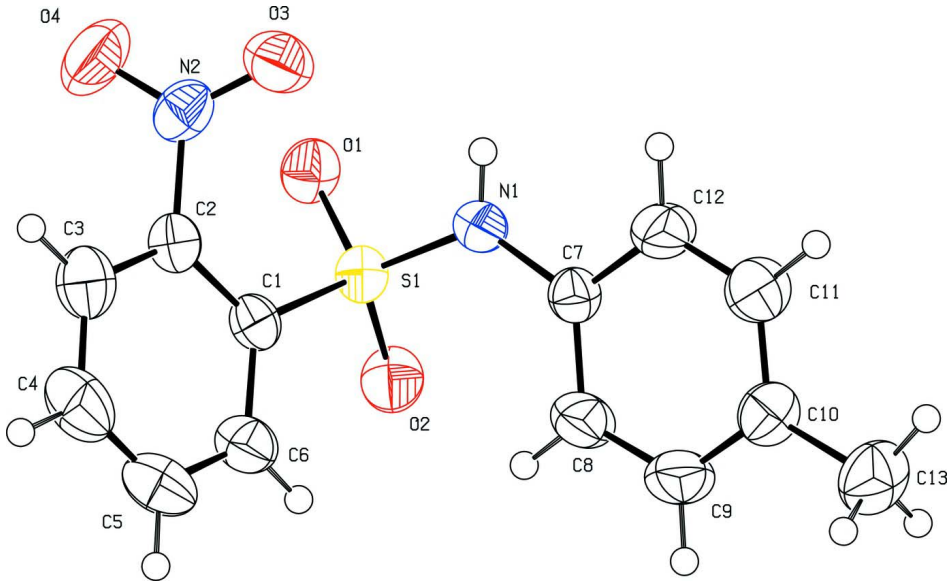

Figure 1

Molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbiterary radius.

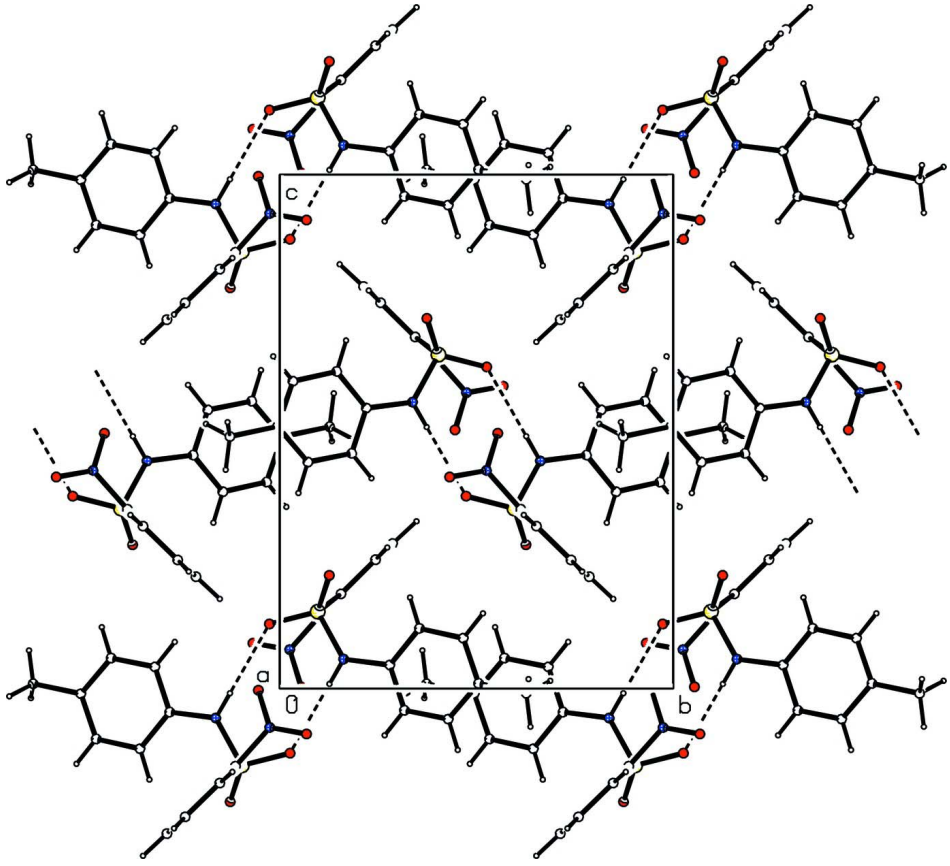

Figure 2

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

N-(4-Methylphenyl)-2-nitrobenzenesulfonamide

Crystal data

$C_{13}H_{12}N_2O_4S$
$M_r = 292.32$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.2787\ (5)$ Å
$b = 11.2017\ (7)$ Å
$c = 14.6435\ (8)$ Å
$\beta = 91.116\ (5)°$
$V = 1357.72\ (14)$ Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.430$ Mg m^{-3}
Melting point: 385 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2975 reflections
$\theta = 2.9–27.8°$
$\mu = 0.25$ mm^{-1}
$T = 295$ K
Prism, yellow
0.48 × 0.40 × 0.40 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)
$T_{min} = 0.888$, $T_{max} = 0.906$
5351 measured reflections
2766 independent reflections
2353 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.1°$
$h = -10\rightarrow10$
$k = -14\rightarrow11$
$l = -18\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.06$
2766 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
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.5176P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3224 (2)	0.15569 (16)	0.18420 (11)	0.0364 (4)
C2	0.1752 (2)	0.11277 (16)	0.15102 (12)	0.0401 (4)
C3	0.0303 (2)	0.1524 (2)	0.18391 (16)	0.0571 (5)
H3	−0.0667	0.1204	0.1622	0.068*
C4	0.0316 (3)	0.2405 (2)	0.24967 (17)	0.0669 (7)
H4	−0.0655	0.2687	0.2722	0.080*
C5	0.1747 (3)	0.2869 (2)	0.28221 (15)	0.0626 (6)
H5	0.1741	0.3476	0.3255	0.075*
C6	0.3206 (3)	0.24352 (19)	0.25080 (13)	0.0485 (5)
H6	0.4174	0.2735	0.2745	0.058*
C7	0.6251 (2)	0.27979 (16)	0.04577 (12)	0.0373 (4)
C8	0.6677 (3)	0.35516 (19)	0.11701 (14)	0.0565 (6)
H8	0.6533	0.3314	0.1772	0.068*
C9	0.7320 (3)	0.46652 (19)	0.09806 (15)	0.0588 (6)
H9	0.7602	0.5167	0.1464	0.071*
C10	0.7555 (2)	0.50554 (18)	0.01005 (14)	0.0480 (5)
C11	0.7119 (3)	0.4285 (2)	−0.05929 (14)	0.0610 (6)
H11	0.7252	0.4525	−0.1195	0.073*
C12	0.6490 (3)	0.3172 (2)	−0.04227 (13)	0.0541 (5)
H12	0.6225	0.2668	−0.0908	0.065*
C13	0.8262 (3)	0.6268 (2)	−0.00946 (18)	0.0665 (6)
H13A	0.9318	0.6326	0.0185	0.100*
H13B	0.7576	0.6875	0.0150	0.100*
H13C	0.8341	0.6374	−0.0743	0.100*
N1	0.5608 (2)	0.16250 (14)	0.05726 (10)	0.0425 (4)
H1N	0.535 (3)	0.1265 (18)	0.0090 (12)	0.051*
N2	0.16650 (18)	0.02534 (14)	0.07570 (12)	0.0465 (4)
O1	0.48358 (16)	−0.02622 (11)	0.12570 (9)	0.0459 (3)
O2	0.62478 (16)	0.12592 (13)	0.22019 (9)	0.0503 (4)
O3	0.22587 (19)	0.05430 (15)	0.00333 (10)	0.0592 (4)
O4	0.0967 (2)	−0.06803 (16)	0.08834 (14)	0.0770 (5)
S1	0.51096 (5)	0.09646 (4)	0.14959 (3)	0.03688 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0385 (9)	0.0381 (9)	0.0326 (8)	0.0024 (7)	0.0034 (7)	0.0054 (7)
C2	0.0390 (9)	0.0412 (10)	0.0403 (9)	−0.0001 (7)	0.0037 (7)	0.0042 (8)
C3	0.0390 (10)	0.0669 (14)	0.0655 (13)	0.0028 (10)	0.0086 (9)	0.0010 (11)
C4	0.0542 (13)	0.0830 (17)	0.0643 (14)	0.0177 (12)	0.0183 (11)	−0.0067 (13)
C5	0.0699 (15)	0.0674 (15)	0.0509 (12)	0.0141 (12)	0.0108 (11)	−0.0132 (11)
C6	0.0513 (11)	0.0551 (12)	0.0390 (10)	0.0018 (9)	0.0013 (8)	−0.0033 (9)
C7	0.0365 (9)	0.0372 (9)	0.0385 (9)	0.0009 (7)	0.0024 (7)	0.0010 (7)
C8	0.0853 (16)	0.0478 (11)	0.0368 (10)	−0.0110 (11)	0.0101 (10)	−0.0022 (9)
C9	0.0868 (16)	0.0428 (11)	0.0470 (11)	−0.0104 (11)	0.0053 (11)	−0.0089 (9)

C10	0.0519 (11)	0.0392 (10)	0.0528 (11)	0.0008 (8)	−0.0014 (9)	0.0059 (9)
C11	0.0830 (16)	0.0606 (13)	0.0391 (10)	−0.0181 (12)	−0.0052 (10)	0.0124 (10)
C12	0.0717 (14)	0.0545 (12)	0.0359 (10)	−0.0158 (10)	−0.0054 (9)	0.0009 (9)
C13	0.0814 (17)	0.0459 (12)	0.0720 (15)	−0.0096 (11)	−0.0013 (13)	0.0118 (11)
N1	0.0512 (9)	0.0412 (9)	0.0353 (8)	−0.0074 (7)	0.0066 (7)	−0.0040 (6)
N2	0.0368 (8)	0.0437 (9)	0.0587 (10)	−0.0018 (7)	−0.0039 (7)	−0.0016 (8)
O1	0.0465 (7)	0.0362 (7)	0.0550 (8)	0.0030 (5)	0.0038 (6)	0.0047 (6)
O2	0.0429 (7)	0.0616 (9)	0.0462 (7)	−0.0010 (6)	−0.0077 (6)	0.0073 (6)
O3	0.0638 (9)	0.0675 (10)	0.0464 (8)	0.0009 (8)	0.0024 (7)	−0.0072 (7)
O4	0.0710 (11)	0.0558 (10)	0.1045 (14)	−0.0252 (8)	0.0067 (10)	−0.0084 (9)
S1	0.0344 (2)	0.0384 (3)	0.0378 (2)	0.00117 (17)	0.00018 (17)	0.00418 (18)

Geometric parameters (Å, º)

C1—C6	1.386 (3)	C9—C10	1.378 (3)
C1—C2	1.389 (3)	C9—H9	0.9300
C1—S1	1.7786 (18)	C10—C11	1.375 (3)
C2—C3	1.375 (3)	C10—C13	1.508 (3)
C2—N2	1.476 (2)	C11—C12	1.376 (3)
C3—C4	1.378 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.371 (4)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.388 (3)	C13—H13C	0.9600
C5—H5	0.9300	N1—S1	1.6022 (16)
C6—H6	0.9300	N1—H1N	0.837 (15)
C7—C12	1.374 (3)	N2—O4	1.211 (2)
C7—C8	1.382 (3)	N2—O3	1.221 (2)
C7—N1	1.429 (2)	O1—S1	1.4349 (14)
C8—C9	1.386 (3)	O2—S1	1.4240 (14)
C8—H8	0.9300		
C6—C1—C2	118.06 (17)	C11—C10—C9	116.84 (19)
C6—C1—S1	119.16 (14)	C11—C10—C13	121.48 (19)
C2—C1—S1	122.68 (14)	C9—C10—C13	121.7 (2)
C3—C2—C1	122.09 (19)	C10—C11—C12	121.98 (19)
C3—C2—N2	116.46 (17)	C10—C11—H11	119.0
C1—C2—N2	121.40 (16)	C12—C11—H11	119.0
C2—C3—C4	118.7 (2)	C7—C12—C11	120.55 (19)
C2—C3—H3	120.6	C7—C12—H12	119.7
C4—C3—H3	120.6	C11—C12—H12	119.7
C5—C4—C3	120.6 (2)	C10—C13—H13A	109.5
C5—C4—H4	119.7	C10—C13—H13B	109.5
C3—C4—H4	119.7	H13A—C13—H13B	109.5
C4—C5—C6	120.2 (2)	C10—C13—H13C	109.5
C4—C5—H5	119.9	H13A—C13—H13C	109.5
C6—C5—H5	119.9	H13B—C13—H13C	109.5
C1—C6—C5	120.2 (2)	C7—N1—S1	128.79 (13)

C1—C6—H6	119.9	C7—N1—H1N	115.7 (15)
C5—C6—H6	119.9	S1—N1—H1N	115.1 (15)
C12—C7—C8	118.87 (18)	O4—N2—O3	124.30 (19)
C12—C7—N1	116.85 (16)	O4—N2—C2	118.40 (18)
C8—C7—N1	124.26 (16)	O3—N2—C2	117.25 (16)
C7—C8—C9	119.45 (19)	O2—S1—O1	119.88 (8)
C7—C8—H8	120.3	O2—S1—N1	109.16 (8)
C9—C8—H8	120.3	O1—S1—N1	106.17 (8)
C10—C9—C8	122.30 (19)	O2—S1—C1	106.18 (8)
C10—C9—H9	118.8	O1—S1—C1	106.94 (8)
C8—C9—H9	118.8	N1—S1—C1	108.05 (8)
C6—C1—C2—C3	−1.7 (3)	C8—C7—C12—C11	−1.0 (3)
S1—C1—C2—C3	174.45 (16)	N1—C7—C12—C11	−179.4 (2)
C6—C1—C2—N2	175.75 (16)	C10—C11—C12—C7	1.1 (4)
S1—C1—C2—N2	−8.0 (2)	C12—C7—N1—S1	−176.43 (16)
C1—C2—C3—C4	2.3 (3)	C8—C7—N1—S1	5.2 (3)
N2—C2—C3—C4	−175.36 (19)	C3—C2—N2—O4	−58.7 (2)
C2—C3—C4—C5	−0.6 (4)	C1—C2—N2—O4	123.6 (2)
C3—C4—C5—C6	−1.6 (4)	C3—C2—N2—O3	118.8 (2)
C2—C1—C6—C5	−0.4 (3)	C1—C2—N2—O3	−58.9 (2)
S1—C1—C6—C5	−176.78 (16)	C7—N1—S1—O2	−38.49 (19)
C4—C5—C6—C1	2.1 (3)	C7—N1—S1—O1	−169.02 (16)
C12—C7—C8—C9	0.5 (3)	C7—N1—S1—C1	76.55 (18)
N1—C7—C8—C9	178.8 (2)	C6—C1—S1—O2	18.56 (17)
C7—C8—C9—C10	−0.1 (4)	C2—C1—S1—O2	−157.61 (15)
C8—C9—C10—C11	0.1 (4)	C6—C1—S1—O1	147.65 (15)
C8—C9—C10—C13	−179.8 (2)	C2—C1—S1—O1	−28.52 (17)
C9—C10—C11—C12	−0.6 (4)	C6—C1—S1—N1	−98.44 (16)
C13—C10—C11—C12	179.2 (2)	C2—C1—S1—N1	85.40 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.84 (2)	2.27 (2)	3.099 (2)	169 (2)

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