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N-(4-Methylphenylsulfonyl)succinamic acid

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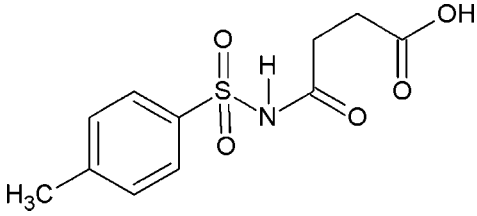
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.110; data-to-parameter ratio = 15.3.

In the crystal structure of the title compound, $C_{11}H_{13}NO_5S$, the amide C=O and the carboxyl C=O groups of the acid segment orient themselves away from each other. The dihedral angle between the benzene ring and the amide group is 69.0 (2)°. In the crystal, N—H···O and O—H···O hydrogen bonds link the molecules into layers parallel to the *bc* plane.

Related literature

For our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2000); Saraswathi *et al.* (2011), of *N*-chloroarylamides, see: Gowda & Rao (1989); Jyothi & Gowda (2004) and of *N*-bromoarylsulfonamides, see: Gowda & Mahadevappa (1983); Usha & Gowda (2006).



Experimental

Crystal data

$C_{11}H_{13}NO_5S$
M_r = 271.28
Monoclinic, $P2_1/c$
a = 10.2496 (9) Å
b = 17.041 (2) Å
c = 7.4721 (6) Å
β = 101.909 (9)°
V = 1277.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 293 K
0.48 × 0.32 × 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} = 0.883, T_{max} = 0.959
4739 measured reflections
2597 independent reflections
2107 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.110
S = 1.12
2597 reflections
170 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 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}	0.83 (2)	2.14 (2)	2.948 (2)	164 (2)
O5—H5*O*···O4^{ii}	0.83 (2)	1.83 (2)	2.663 (3)	178 (3)

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

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: RZ2761).

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supplementary materials

Acta Cryst. (2012). E**68**, o1885 [doi:10.1107/S1600536812023276]

N-(4-Methylphenylsulfonyl)succinamic acid

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Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000; Saraswathi *et al.*, 2011); *N*-chloroarylsulfonamides (Gowda & Rao, 1989; Jyothi & Gowda, 2004) and *N*-bromo-aryl- sulfonamides (Gowda & Mahadevappa, 1983; Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(4-methylphenylsulfonyl)succinamic acid has been determined (Fig. 1). The conformations of the N—H and C=O bonds in the amide segment are *anti* to each other. Further, the amide C═O and the carboxyl C═O of the acid segment orient themselves away from each other, in contrast to the *anti* conformation observed between the the amide oxygen and the carboxyl oxygen in *N*-(4-methylphenyl)-succinamic acid (I) (Saraswathi *et al.*, 2011). But both the amide oxygen and the carboxyl oxygen are *anti* to the H atoms on the adjacent –CH_2 groups, in both the compounds.

In the title compound, the C═O and O—H bonds of the acid group are in *syn* position to each other, similar to that observed in (I). The molecule is bent at the S-atom with the C1–S1–N1–C7 torsion angle of 79.2 (1)°. Further, the dihedral angle between the phenyl ring and the amide group is 69.0 (2)°. In the crystal, the pairs of O—H···O and N—H···O intermolecular hydrogen bonds link the molecules into layers parallel to the *bc* plane (Table 1, Fig. 2).

Experimental

Succinic anhydride (0.015 mole) and 4-dimethylaminopyridine (0.01 mole) were added to a solution of *p*-toluene-sulfonamide (0.01 mole) in dichloromethane. The reaction mixture was strirred for 18 h at room temperature and set aside for completion of the reaction. The reaction mixture was concentrated to dryness. The resultant title compound was washed with dilute HCl and then with water thoroughly, to remove the unreacted base and the succinic anhydride. It was recrystallized to constant melting point from ethyl acetate (173–175 °C). The purity of the compound was checked and characterized by its infrared spectrum. Prism-like colourless single crystals used in X-ray diffraction studies were grown by slow evaporation of an ethyl acetate solution at room temperature.

Refinement

The H atoms of the NH group and the OH group were located in a difference Fourier map and later restrained to the distances of N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, respectively. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å and methylene C—H = 0.97 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).

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).

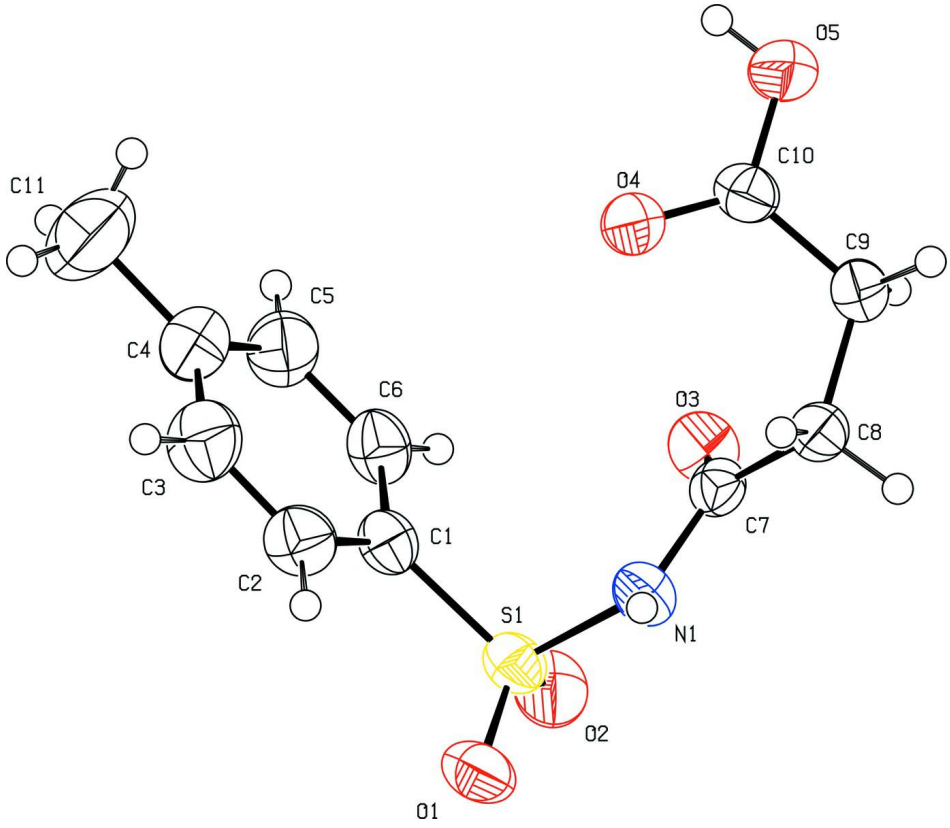

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

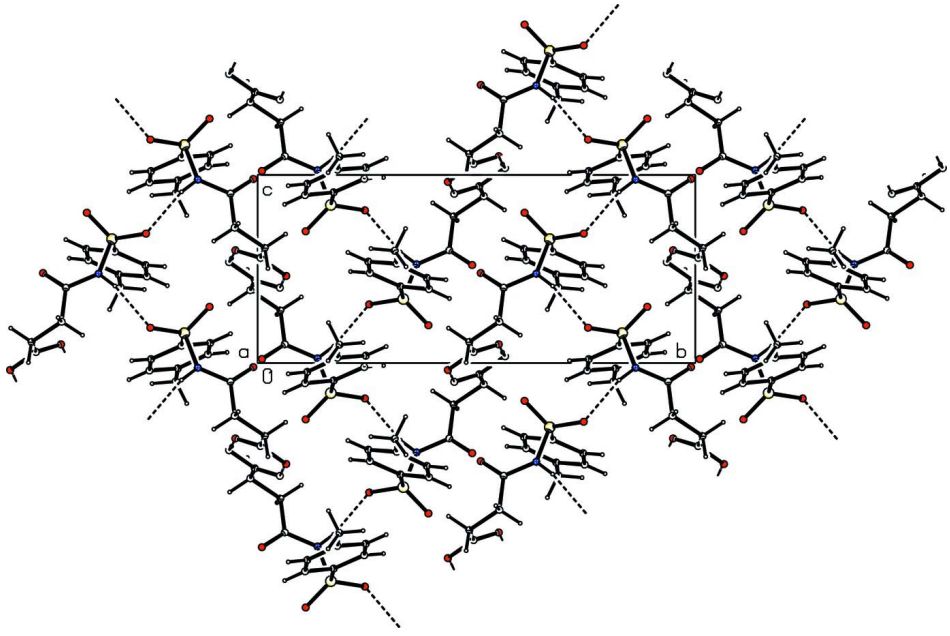

Figure 2

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

N-(4-Methylphenylsulfonyl)succinamic acid

Crystal data

$C_{11}H_{13}NO_5S$
$M_r = 271.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.2496$ (9) Å
$b = 17.041$ (2) Å
$c = 7.4721$ (6) Å
$\beta = 101.909$ (9)°
$V = 1277.0$ (2) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2495 reflections
$\theta = 3.0$–$27.6°$
$\mu = 0.27$ mm^{-1}
$T = 293$ K
Prism, colourless
0.48 × 0.32 × 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)
$T_{min} = 0.883$, $T_{max} = 0.959$
4739 measured reflections
2597 independent reflections
2107 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
$h = -12 \rightarrow 11$
$k = -21 \rightarrow 8$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.110$
$S = 1.12$
2597 reflections
170 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
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.9166P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Experimental. Absorption correction: 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	0.1649 (2)	0.82620 (14)	0.0924 (3)	0.0398 (5)

C2	0.1077 (3)	0.75638 (15)	0.0207 (4)	0.0507 (6)
H2	0.1604	0.7126	0.0132	0.061*
C3	−0.0291 (3)	0.75296 (17)	−0.0394 (4)	0.0625 (7)
H3	−0.0682	0.7063	−0.0881	0.075*
C4	−0.1092 (3)	0.81770 (18)	−0.0286 (4)	0.0607 (7)
C5	−0.0496 (3)	0.88660 (16)	0.0469 (4)	0.0582 (7)
H5	−0.1025	0.9301	0.0572	0.070*
C6	0.0864 (3)	0.89152 (14)	0.1068 (4)	0.0482 (6)
H6	0.1255	0.9381	0.1563	0.058*
C7	0.3668 (2)	0.93791 (13)	−0.0993 (3)	0.0349 (5)
C8	0.4086 (2)	0.94773 (14)	−0.2799 (3)	0.0394 (5)
H8A	0.3879	0.9001	−0.3514	0.047*
H8B	0.5042	0.9557	−0.2582	0.047*
C9	0.3388 (2)	1.01660 (14)	−0.3874 (3)	0.0439 (6)
H9A	0.3582	1.0638	−0.3143	0.053*
H9B	0.3743	1.0236	−0.4971	0.053*
C10	0.1912 (2)	1.00645 (14)	−0.4408 (3)	0.0419 (5)
C11	−0.2586 (3)	0.8130 (2)	−0.0975 (6)	0.0995 (13)
H11A	−0.2856	0.7590	−0.1091	0.119*
H11B	−0.2822	0.8382	−0.2147	0.119*
H11C	−0.3029	0.8389	−0.0126	0.119*
N1	0.3902 (2)	0.86326 (11)	−0.0248 (2)	0.0387 (4)
H1N	0.404 (3)	0.8261 (12)	−0.090 (3)	0.046*
O1	0.39301 (18)	0.75502 (11)	0.1880 (2)	0.0565 (5)
O2	0.37311 (18)	0.89014 (11)	0.2992 (2)	0.0558 (5)
O3	0.32175 (17)	0.99012 (10)	−0.0214 (2)	0.0479 (4)
O4	0.13237 (16)	0.94917 (10)	−0.4003 (3)	0.0552 (5)
O5	0.13192 (19)	1.06525 (12)	−0.5367 (3)	0.0672 (6)
H5O	0.0496 (18)	1.0596 (19)	−0.557 (4)	0.081*
S1	0.33860 (6)	0.83267 (4)	0.15920 (7)	0.04120 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0465 (13)	0.0405 (13)	0.0342 (11)	−0.0018 (10)	0.0130 (9)	0.0004 (10)
C2	0.0567 (15)	0.0388 (13)	0.0565 (15)	0.0028 (12)	0.0116 (12)	−0.0042 (12)
C3	0.0639 (18)	0.0487 (16)	0.0723 (19)	−0.0113 (14)	0.0080 (14)	−0.0089 (14)
C4	0.0482 (15)	0.0634 (18)	0.0701 (18)	−0.0024 (13)	0.0112 (13)	0.0047 (15)
C5	0.0537 (16)	0.0479 (15)	0.0771 (19)	0.0076 (13)	0.0230 (14)	0.0025 (14)
C6	0.0562 (15)	0.0359 (13)	0.0564 (15)	−0.0021 (11)	0.0209 (12)	−0.0030 (11)
C7	0.0315 (11)	0.0392 (12)	0.0328 (11)	−0.0044 (9)	0.0038 (9)	−0.0022 (9)
C8	0.0365 (12)	0.0479 (13)	0.0336 (11)	−0.0005 (10)	0.0072 (9)	0.0029 (10)
C9	0.0405 (12)	0.0479 (14)	0.0430 (12)	−0.0051 (11)	0.0082 (10)	0.0080 (11)
C10	0.0464 (13)	0.0400 (13)	0.0376 (12)	0.0007 (11)	0.0047 (10)	0.0042 (10)
C11	0.054 (2)	0.098 (3)	0.140 (4)	−0.0050 (19)	0.003 (2)	−0.001 (3)
N1	0.0485 (11)	0.0360 (10)	0.0341 (10)	0.0027 (9)	0.0142 (8)	−0.0011 (8)
O1	0.0609 (11)	0.0531 (11)	0.0559 (11)	0.0110 (9)	0.0129 (9)	0.0202 (9)
O2	0.0649 (11)	0.0689 (12)	0.0326 (8)	−0.0113 (10)	0.0075 (8)	−0.0062 (8)
O3	0.0580 (10)	0.0423 (10)	0.0467 (9)	0.0046 (8)	0.0181 (8)	−0.0041 (8)
O4	0.0420 (9)	0.0466 (10)	0.0718 (12)	−0.0054 (8)	−0.0001 (8)	0.0162 (9)

O5	0.0451 (10)	0.0579 (12)	0.0924 (15)	0.0011 (9)	0.0001 (10)	0.0301 (11)
S1	0.0478 (3)	0.0445 (3)	0.0315 (3)	−0.0001 (3)	0.0087 (2)	0.0046 (2)

Geometric parameters (Å, º)

C1—C2	1.385 (3)	C8—H8A	0.9700
C1—C6	1.390 (3)	C8—H8B	0.9700
C1—S1	1.750 (2)	C9—C10	1.493 (3)
C2—C3	1.382 (4)	C9—H9A	0.9700
C2—H2	0.9300	C9—H9B	0.9700
C3—C4	1.387 (4)	C10—O4	1.218 (3)
C3—H3	0.9300	C10—O5	1.307 (3)
C4—C5	1.388 (4)	C11—H11A	0.9600
C4—C11	1.514 (4)	C11—H11B	0.9600
C5—C6	1.377 (4)	C11—H11C	0.9600
C5—H5	0.9300	N1—S1	1.6559 (19)
C6—H6	0.9300	N1—H1N	0.828 (16)
C7—O3	1.206 (3)	O1—S1	1.4349 (19)
C7—N1	1.390 (3)	O2—S1	1.4234 (18)
C7—C8	1.507 (3)	O5—H5O	0.832 (18)
C8—C9	1.516 (3)		
C2—C1—C6	120.8 (2)	H8A—C8—H8B	107.9
C2—C1—S1	119.28 (19)	C10—C9—C8	113.14 (19)
C6—C1—S1	119.88 (19)	C10—C9—H9A	109.0
C3—C2—C1	118.8 (2)	C8—C9—H9A	109.0
C3—C2—H2	120.6	C10—C9—H9B	109.0
C1—C2—H2	120.6	C8—C9—H9B	109.0
C2—C3—C4	121.3 (3)	H9A—C9—H9B	107.8
C2—C3—H3	119.3	O4—C10—O5	123.6 (2)
C4—C3—H3	119.3	O4—C10—C9	123.6 (2)
C3—C4—C5	118.7 (3)	O5—C10—C9	112.9 (2)
C3—C4—C11	120.5 (3)	C4—C11—H11A	109.5
C5—C4—C11	120.8 (3)	C4—C11—H11B	109.5
C6—C5—C4	121.0 (2)	H11A—C11—H11B	109.5
C6—C5—H5	119.5	C4—C11—H11C	109.5
C4—C5—H5	119.5	H11A—C11—H11C	109.5
C5—C6—C1	119.3 (2)	H11B—C11—H11C	109.5
C5—C6—H6	120.3	C7—N1—S1	124.26 (16)
C1—C6—H6	120.3	C7—N1—H1N	120.1 (18)
O3—C7—N1	122.2 (2)	S1—N1—H1N	111.6 (18)
O3—C7—C8	123.9 (2)	C10—O5—H5O	111 (2)
N1—C7—C8	113.78 (19)	O2—S1—O1	119.62 (11)
C7—C8—C9	111.76 (19)	O2—S1—N1	108.68 (10)
C7—C8—H8A	109.3	O1—S1—N1	103.54 (10)
C9—C8—H8A	109.3	O2—S1—C1	109.68 (11)
C7—C8—H8B	109.3	O1—S1—C1	109.01 (11)
C9—C8—H8B	109.3	N1—S1—C1	105.27 (10)
C6—C1—C2—C3	1.0 (4)	C8—C9—C10—O4	0.8 (3)

S1—C1—C2—C3	−177.0 (2)	C8—C9—C10—O5	−178.6 (2)
C1—C2—C3—C4	−0.2 (4)	O3—C7—N1—S1	10.0 (3)
C2—C3—C4—C5	−0.9 (5)	C8—C7—N1—S1	−172.55 (15)
C2—C3—C4—C11	179.2 (3)	C7—N1—S1—O2	−48.4 (2)
C3—C4—C5—C6	1.3 (4)	C7—N1—S1—O1	−176.60 (18)
C11—C4—C5—C6	−178.8 (3)	C7—N1—S1—C1	69.0 (2)
C4—C5—C6—C1	−0.5 (4)	C2—C1—S1—O2	−153.06 (19)
C2—C1—C6—C5	−0.7 (4)	C6—C1—S1—O2	28.8 (2)
S1—C1—C6—C5	177.4 (2)	C2—C1—S1—O1	−20.3 (2)
O3—C7—C8—C9	−23.2 (3)	C6—C1—S1—O1	161.55 (18)
N1—C7—C8—C9	159.36 (19)	C2—C1—S1—N1	90.2 (2)
C7—C8—C9—C10	−63.7 (3)	C6—C1—S1—N1	−87.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.83 (2)	2.14 (2)	2.948 (2)	164 (2)
O5—H5*O*···O4^{ii}	0.83 (2)	1.83 (2)	2.663 (3)	178 (3)

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