IUCrData

ISSN 2414-3146

4-Bromo-*N*-(4-fluorophenyl)benzenesulfonamide

Vinola Z. Rodrigues,[a] P. A. Suchetan,[b] L. Saritha,[a] N. K. Lokanath[c] and S. Naveen[d]*

[a]Department of PG Studies and Research in Chemistry, St Aloysius College, Mangalore, India, [b]Department of Chemistry, University College of Science, Tumkur University, Tumkur 572 103, India, [c]Department of Studies in Physics, University of Mysore, Manasagangotri, Mysuru-6, India, and [d]Institution of Excellence, University of Mysore, Manasagangotri, Mysuru-6, India. *Correspondence e-mail: naveen@ioe.uni-mysore.ac.in

Received 21 July 2016
Accepted 4 August 2016

Edited by A. J. Lough, University of Toronto, Canada

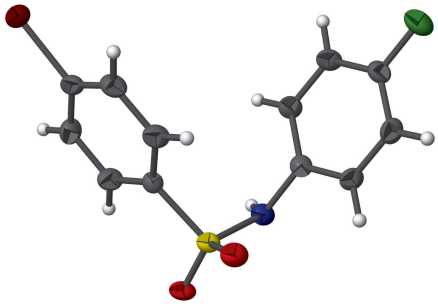
Keywords: crystal structure; sulfonamides; N—H···O hydrogen bonds; F···F contacts.

CCDC reference: 1497562

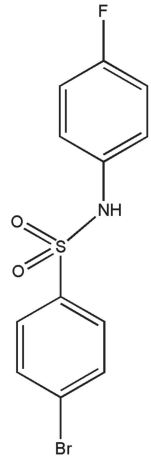
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_{12}H_9BrFNO_2S$, is U-shaped with the central C—S—N—C fragment having a torsion angle of 68.4 (3)° and a dihedral angle between the planes of the two benzene rings of 41.17 (19)°. The crystal structure features strong N—H···O hydrogen bonds between the molecules, forming infinite one-dimensional *C*(4) chains along [001]. These chains are interconnected *via* short F···F contacts [F···F = 2.868 (4) Å], forming a one-dimensional ribbon-like architecture.

3D view



Chemical scheme



Structure description

Sulfonamide drugs were the first among the chemotherapeutic agents to be used for the curing and prevention of bacterial infection in human beings (Shiva Prasad *et al.*, 2011). They play a vital role as a key constituent in a number of biologically active molecules. To date, sulfonamides have been known to exhibit a wide variety of biological activities, such as antibacterial (Subhakara Reddy *et al.*, 2012), insecticidal (Himel *et al.*, 1971), antifungal (Hanafy *et al.*, 2007), antihepatitis (Zhao *et al.*, 2010), anti-inflammatory (Küçükgüzel *et al.*, 2013), antitumour (Ghorab *et al.*, 2011), anticancer (Al-Said *et al.*, 2011), anti-HIV (Sahu *et al.*, 2007) and antitubercular activities (Vora & Mehta, 2012). In recent years, extensive research studies have been carried out on the synthesis and evaluation of the pharmacological activities of molecules containing a sulfonamide moiety, and important pharmacophores have been reported (Mohan *et al.*, 2013). With this in mind and in our continued efforts for understanding the structures of *N*-(4-substituted phenyl)-4-bromobenzenesulfonamides (Rodrigues *et al.*, 2015, 2016), we report herein the crystal structure of 4-bromo-*N*-(4-fluorophenyl)benzenesulfonamide, (I).

The molecule of (I) (Fig. 1) is U-shaped, with the central C4—S1—N1—C7 fragment having a torsion angle of 68.4 (3)°. The dihedral angle between the planes of the two

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.90 (2)	2.03 (2)	2.911 (4)	167 (4)

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

benzene rings is 41.17 (19)°. In comparison, the dihedral angles between the planes of the two benzene rings in the reported structures of 4-bromo-*N*-(4-bromophenyl)benzenesulfonamide, (II) (Rodrigues *et al.*, 2015), and 4-bromo-*N*-(4-nitrophenyl)benzenesulfonamide, (III) (Rodrigues *et al.*, 2016), are slightly less than that in (I), with values of 38.5 (2)° in (II) and 32.6 (6)° in (III).

The crystal structure of (I) features strong N1—H1···O2^{i} hydrogen bonds (Table 1 and Fig. 2) between the molecules, forming infinite one-dimensional $C(4)$ chains along [001]. These chains are interconnected *via* F···F($-x$, $-y$, $z+\frac{1}{2}$) contacts [F···F = 2.868 (4) Å], forming a one-dimensional ribbon-like architecture (Fig. 2). In contrast, in (II) and (III), three-dimensional supramolecular architectures are present as a result of N—H···O(S) chains, structure-directing C—H···O interactions and other weak interactions of the types Br···Br [in (II)] and Br···O$_{nitro}$ [in (III)].

Synthesis and crystallization

Compound (I) was prepared according to the literature method of Rodrigues *et al.* (2015). The purity of the compound was checked by determining its melting point (m.p. 400 K). Prismatic single crystals of (I) used for X-ray diffraction study were obtained by slow evaporation of an ethanolic solution of (I) at room temperature.

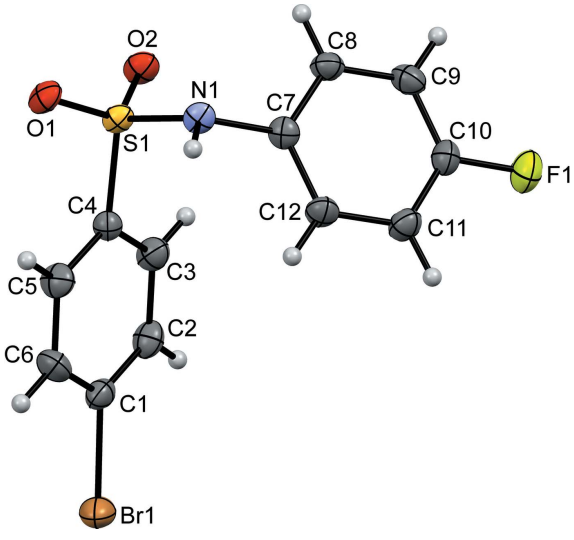


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_9BrFNO_2S$
M_r	330.17
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	296
a, b, c (Å)	19.7608 (6), 12.4156 (4), 5.0776 (2)
V (Å^3)	1245.75 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.14
Crystal size (mm)	0.27 × 0.24 × 0.19
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.238, 0.311
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5649, 1688, 1621
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.583
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.083, 1.03
No. of reflections	1688
No. of parameters	167
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.36
Absolute structure	Flack (1983), 547 Friedel pairs
Absolute structure parameter	0.01 (3)

Computer programs: *APEX2*, *SAINT-Plus* and *XPREP* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Two reflections with bad agreement between F_o and F_c, *viz.* 110 and 120, were omitted from the final refinement.

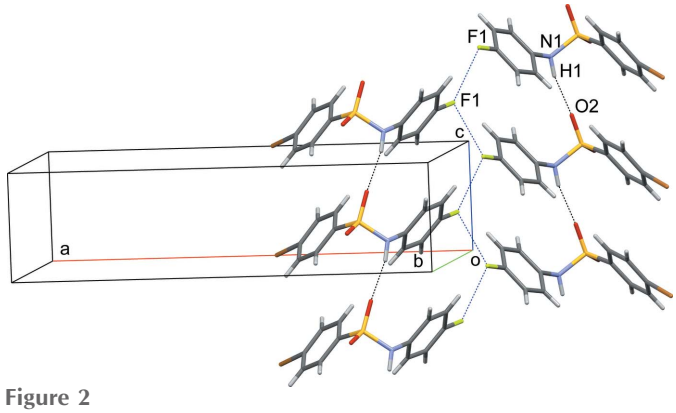


Figure 2
Crystal packing of the title compound, showing N—H···O hydrogen bonds as dotted lines. The F···F contacts between hydrogen-bonded chains are also shown as dotted lines.

Acknowledgements

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysore, for providing the single-crystal X-ray diffraction data. VZR is thankful to the University Grants Commission, Delhi, for financial assistance under its MRP scheme.

References

Al-Said, M. S., Ghorab, M. M., Al-Dosari, M. S. & Hamed, M. M. (2011). *Eur. J. Med. Chem.* **46**, 201–207.
Bruker (2009). *APEX2*, *SADABS*, *SAINT-Plus* and *XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Ghorab, M. M., Ragab, A. F., Heiba, I. H. & Agha, M. H. (2011). *J. Basic Appl .Chem*. **1**(2), 8–14.
Hanafy, A., Uno, J., Mitani, H., Kang, Y. & Mikami, Y. (2007). *Nippon Ishinkin Gakkai Zasshi*, **48**, 47–50.
Himel, C. M., Aboul-Saad, W. G. & Uk, S. (1971). *J. Agric. Food Chem.* **19**, 1175–1180.
Küçükgüzel, S. G., Coskun, I., Aydın, S., Aktay, G., Gursoy, S., Cevik, O., Ozakpınar, O. B., Ozsavc, D., Sener, A., Kaushik-Basu, N., Basu, A. & Talele, T. T. (2013). *Molecules*, **18**, 3595–3614.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mohan, N. R., Sreenivasa, S., Manojkumar, K. E. & Chakrapani Rao, T. M. (2013). *J. Appl. Chem.* **2**, 722–729.
Reddy, N. S., Rao, A. S., Chari, M. A., Kumar, V. R., Jyothy, V. & Himabindu, V. (2012). *J. Chem. Sci.* **124**, 723–730.
Rodrigues, V. Z., Naveen, S., Lokanath, N. K. & Suchetan, P. A. (2016). *IUCrData*, **1**, x160631.
Rodrigues, V. Z., Snehala, Naveen, S., Lokanath, N. K. & Suchetan, P. A. (2015). *Der Pharma Chem.* **7**, 299–307.
Sahu, K. K., Ravichandran, V., Mourya, V. K. & Agrawal, R. K. (2007). *Med. Chem. Res.* **15**, 418–430.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shiva Prasad, K., Shiva Kumara, L., Vinay, K. B., Chandra Shekar, S., Jayalakshmi, B. & Revanasiddappa, H. D. (2011). *Int. J. Chem. Res.* **2**, 1–6.
Vora, P. J. & Mehta, A. G. (2012). *IOSR J. Appl. Chem.* **1**, 34–39.
Zhao, Y.-F., Feng, R.-L., Liu, Y.-J., Zhang, Y.-K. & Gong, P. (2010). *Chem. Res. Chin. Univ.* **26**, 272–277.

full crystallographic data

IUCrData (2016). **1**, x161256 [doi:10.1107/S2414314616012566]

4-Bromo-*N*-(4-fluorophenyl)benzenesulfonamide

Vinola Z. Rodrigues, P. A. Suchetan, L. Saritha, N. K. Lokanath and S. Naveen

4-Bromo-*N*-(4-fluorophenyl)benzenesulfonamide

Crystal data

$C_{12}H_9BrFNO_2S$
M_r = 330.17
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 19.7608 (6) Å
b = 12.4156 (4) Å
c = 5.0776 (2) Å
V = 1245.75 (7) Å^3
Z = 4
F(000) = 656
Prism
D_x = 1.760 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 137 reflections
θ = 5.7–64.1°
μ = 6.14 mm^{-1}
T = 296 K
Prism, colourless
0.27 × 0.24 × 0.19 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.238, T_{max} = 0.311
5649 measured reflections
1688 independent reflections
1621 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 64.1°, θ_{min} = 5.7°
h = −22→22
k = −14→12
l = −5→5
1 standard reflections every 1 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.083
S = 1.03
1688 reflections
167 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
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983), 547 Friedel pairs
Absolute structure parameter: 0.01 (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 $F^2 > 2\sigma(F^2)$ 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	0.39127 (18)	0.1909 (3)	0.1353 (8)	0.0265 (8)
C2	0.34960 (19)	0.1649 (3)	0.3425 (9)	0.0296 (9)
H2	0.3549	0.0997	0.4302	0.036*
C3	0.2997 (2)	0.2365 (3)	0.4200 (8)	0.0289 (9)
H3	0.2707	0.2196	0.5580	0.035*
C4	0.29376 (18)	0.3337 (3)	0.2885 (7)	0.0217 (8)
C5	0.3370 (2)	0.3607 (4)	0.0829 (8)	0.0300 (10)
H5	0.3328	0.4267	−0.0022	0.036*
C6	0.38626 (18)	0.2882 (3)	0.0067 (10)	0.0326 (9)
H6	0.4157	0.3049	−0.1301	0.039*
C7	0.13069 (18)	0.2839 (3)	0.2275 (7)	0.0227 (8)
C8	0.08360 (19)	0.2754 (3)	0.4302 (7)	0.0266 (9)
H8	0.0739	0.3348	0.5351	0.032*
C9	0.05147 (18)	0.1789 (3)	0.4750 (9)	0.0296 (10)
H9	0.0208	0.1717	0.6129	0.035*
C10	0.0654 (2)	0.0936 (3)	0.3131 (9)	0.0272 (10)
C11	0.1114 (2)	0.0990 (3)	0.1087 (9)	0.0290 (10)
H11	0.1195	0.0398	0.0009	0.035*
C12	0.14518 (19)	0.1957 (3)	0.0700 (8)	0.0276 (9)
H12	0.1776	0.2014	−0.0620	0.033*
N1	0.16334 (17)	0.3861 (3)	0.1795 (6)	0.0221 (7)
O1	0.24522 (13)	0.5284 (2)	0.2820 (5)	0.0270 (6)
O2	0.20711 (16)	0.4034 (2)	0.6338 (6)	0.0283 (7)
F1	0.03373 (12)	−0.0025 (2)	0.3541 (6)	0.0388 (6)
S1	0.22670 (5)	0.42265 (7)	0.3659 (2)	0.0217 (2)
Br1	0.457510 (19)	0.09041 (3)	0.01845 (10)	0.03674 (18)
H1	0.170 (2)	0.393 (3)	0.005 (3)	0.030 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0271 (18)	0.022 (2)	0.031 (2)	−0.0001 (16)	−0.0040 (15)	−0.0053 (19)
C2	0.035 (2)	0.022 (2)	0.032 (2)	−0.0024 (17)	−0.0043 (17)	0.0052 (19)
C3	0.037 (2)	0.025 (2)	0.025 (2)	−0.0022 (17)	0.0048 (15)	0.0017 (17)
C4	0.0228 (17)	0.023 (2)	0.0193 (19)	−0.0017 (16)	−0.0004 (13)	0.0006 (15)
C5	0.036 (2)	0.023 (2)	0.031 (2)	0.0010 (17)	0.0039 (16)	0.0071 (17)

C6	0.0282 (18)	0.037 (2)	0.033 (2)	−0.0012 (16)	0.0095 (18)	−0.001 (2)
C7	0.0263 (19)	0.024 (2)	0.0178 (19)	−0.0003 (16)	−0.0008 (14)	0.0044 (16)
C8	0.0289 (19)	0.027 (2)	0.024 (2)	0.0023 (17)	0.0050 (14)	−0.0029 (16)
C9	0.0265 (17)	0.033 (2)	0.029 (3)	0.0018 (16)	0.0073 (17)	0.002 (2)
C10	0.028 (2)	0.023 (2)	0.030 (3)	−0.0038 (15)	−0.0023 (18)	0.0020 (16)
C11	0.036 (2)	0.026 (2)	0.024 (2)	−0.0017 (16)	0.0017 (17)	−0.0043 (17)
C12	0.0324 (19)	0.029 (2)	0.021 (2)	−0.0022 (15)	0.0053 (15)	−0.0033 (17)
N1	0.0306 (17)	0.0239 (16)	0.0119 (18)	−0.0016 (14)	0.0008 (13)	0.0029 (14)
O1	0.0390 (15)	0.0165 (14)	0.0254 (14)	−0.0004 (12)	0.0021 (11)	0.0033 (11)
O2	0.0375 (17)	0.0278 (17)	0.0195 (16)	−0.0011 (11)	0.0030 (13)	0.0002 (11)
F1	0.0447 (13)	0.0287 (14)	0.0430 (16)	−0.0109 (10)	−0.0002 (12)	0.0024 (13)
S1	0.0288 (5)	0.0207 (5)	0.0156 (4)	−0.0003 (3)	0.0014 (4)	0.0003 (4)
Br1	0.0296 (3)	0.0300 (3)	0.0506 (3)	0.00444 (15)	0.0016 (2)	−0.0070 (3)

Geometric parameters (Å, º)

C1—C2	1.374 (6)	C7—N1	1.445 (5)
C1—C6	1.377 (6)	C8—C9	1.375 (6)
C1—Br1	1.903 (4)	C8—H8	0.9300
C2—C3	1.385 (6)	C9—C10	1.369 (6)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.384 (6)	C10—F1	1.363 (5)
C3—H3	0.9300	C10—C11	1.382 (7)
C4—C5	1.390 (5)	C11—C12	1.388 (6)
C4—S1	1.769 (4)	C11—H11	0.9300
C5—C6	1.382 (6)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.634 (3)
C6—H6	0.9300	N1—H1	0.900 (10)
C7—C12	1.386 (5)	O1—S1	1.428 (3)
C7—C8	1.392 (5)	O2—S1	1.434 (3)
C2—C1—C6	121.7 (4)	C7—C8—H8	120.1
C2—C1—Br1	119.8 (3)	C10—C9—C8	118.8 (4)
C6—C1—Br1	118.5 (3)	C10—C9—H9	120.6
C1—C2—C3	119.6 (4)	C8—C9—H9	120.6
C1—C2—H2	120.2	F1—C10—C9	119.6 (4)
C3—C2—H2	120.2	F1—C10—C11	117.3 (4)
C4—C3—C2	118.9 (4)	C9—C10—C11	123.1 (4)
C4—C3—H3	120.6	C10—C11—C12	117.7 (4)
C2—C3—H3	120.6	C10—C11—H11	121.1
C3—C4—C5	121.4 (4)	C12—C11—H11	121.1
C3—C4—S1	120.0 (3)	C7—C12—C11	120.2 (4)
C5—C4—S1	118.4 (3)	C7—C12—H12	119.9
C6—C5—C4	119.1 (4)	C11—C12—H12	119.9
C6—C5—H5	120.5	C7—N1—S1	119.2 (2)
C4—C5—H5	120.5	C7—N1—H1	108 (3)
C1—C6—C5	119.3 (4)	S1—N1—H1	116 (3)
C1—C6—H6	120.3	O1—S1—O2	120.34 (16)

C5—C6—H6	120.3	O1—S1—N1	106.20 (17)
C12—C7—C8	120.3 (4)	O2—S1—N1	107.23 (17)
C12—C7—N1	120.3 (3)	O1—S1—C4	108.41 (17)
C8—C7—N1	119.4 (4)	O2—S1—C4	107.98 (18)
C9—C8—C7	119.8 (4)	N1—S1—C4	105.81 (17)
C9—C8—H8	120.1		

C6—C1—C2—C3	2.1 (6)	C9—C10—C11—C12	0.6 (7)
Br1—C1—C2—C3	−177.4 (3)	C8—C7—C12—C11	1.8 (6)
C1—C2—C3—C4	−1.0 (6)	N1—C7—C12—C11	−177.0 (4)
C2—C3—C4—C5	−0.4 (6)	C10—C11—C12—C7	−2.1 (6)
C2—C3—C4—S1	175.3 (3)	C12—C7—N1—S1	−101.4 (4)
C3—C4—C5—C6	0.8 (6)	C8—C7—N1—S1	79.8 (4)
S1—C4—C5—C6	−174.9 (3)	C7—N1—S1—O1	−176.5 (3)
C2—C1—C6—C5	−1.7 (6)	C7—N1—S1—O2	−46.7 (3)
Br1—C1—C6—C5	177.8 (3)	C7—N1—S1—C4	68.4 (3)
C4—C5—C6—C1	0.2 (6)	C3—C4—S1—O1	158.8 (3)
C12—C7—C8—C9	0.2 (6)	C5—C4—S1—O1	−25.4 (4)
N1—C7—C8—C9	179.0 (4)	C3—C4—S1—O2	26.9 (4)
C7—C8—C9—C10	−1.8 (6)	C5—C4—S1—O2	−157.3 (3)
C8—C9—C10—F1	−179.7 (4)	C3—C4—S1—N1	−87.7 (3)
C8—C9—C10—C11	1.4 (7)	C5—C4—S1—N1	88.2 (3)
F1—C10—C11—C12	−178.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.90 (2)	2.03 (2)	2.911 (4)	167 (4)

Symmetry code: (i) *x*, *y*, *z*−1.