



Crystal structures of *N*-(3-fluorobenzoyl)benzenesulfonamide and *N*-(3-fluorobenzoyl)-4-methylbenzenesulfonamide

P. A. Suchetan,^{a,*} S. Naveen,^b§ S. N. K. Lokanath,^c H. N. Lakshmikantha,^d
K. S. Srivishnu^d and G. M. Supriya^d

Received 8 February 2016

Accepted 25 February 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

‡ These authors contributed equally.

§ These authors contributed equally.

Keywords: crystal structure; *N*-(arylsulfonyl)arylamides; N—H···O hydrogen bonds; C—H···O interactions; C—H···π interactions.

CCDC references: 1418689; 1418688

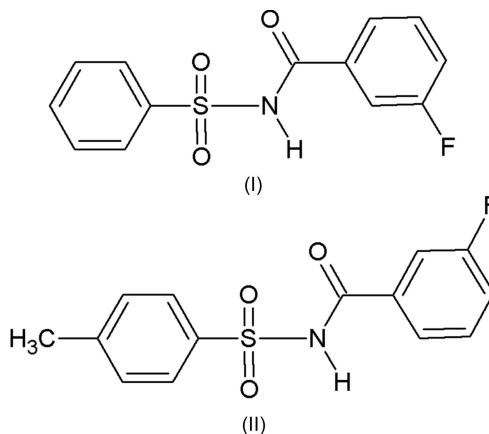
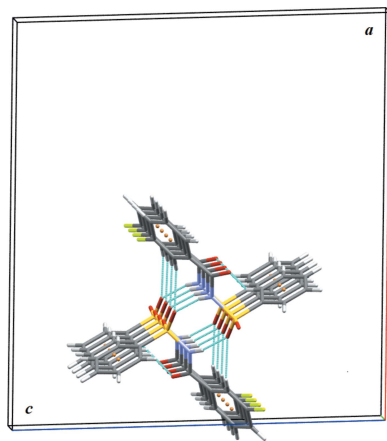
Supporting information: this article has supporting information at journals.iucr.org/e

^aDepartment of Chemistry, University College of Science, Tumkur University, Tumkur 572 103, India, ^bInstitution of Excellence, University of Mysore, Manasagangotri, Mysuru-6, India, ^cDepartment of Physics, University of Mysore, Manasagangotri, Mysuru-6, India, and ^dUniversity College of Science, Tumkur, India. *Correspondence e-mail: pasuchetan@yahoo.co.in

The crystal structures of two *N*-(arylsulfonyl)arylamides, namely *N*-(3-fluorobenzoyl)benzenesulfonamide, C₁₃H₁₀FNO₃S, (I), and *N*-(3-fluorobenzoyl)-4-methylbenzenesulfonamide, C₁₄H₁₂FNO₃S, (II), are described and compared with related structures. The dihedral angle between the benzene rings is 82.73 (10)° in (I) compared to 72.60 (12)° in (II). In the crystal of (I), the molecules are linked by C—H···O and C—H···π interactions, resulting in a three-dimensional grid-like architecture, while C—H···O interactions lead to one-dimensional ribbons in (II). The crystals of both (I) and (II) feature strong but non-structure-directing N—H···O hydrogen bonds with R₂²(8) ring motifs. The structure of (I) also features π–π stacking interactions.

1. Chemical context

N-(Arylsulfonyl)arylamides have received much attention as they constitute an important class of drugs for Alzheimers disease (Hasegawa *et al.*, 2000), antibacterial inhibitors of tRNA synthetases (Banwell *et al.*, 2000), antagonists for angiotensin II (Chang *et al.*, 1994) and as leukotriene D₄-receptors (Musser *et al.*, 1990). Further, *N*-(arylsulfonyl)arylamides are known to be potent antitumour agents against a broad spectrum of human tumour xenografts (colon, lung, breast, ovary and prostate) in nude mice (Mader *et al.*, 2005). As part of our ongoing work on the synthesis and crystal structures of this class of compound (Gowda *et al.*, 2009a,b; Sreenivasa *et al.*, 2014; Suchetan *et al.*, 2010, 2012), compounds (I) and (II) were synthesized and their crystal structures were determined.



OPEN ACCESS

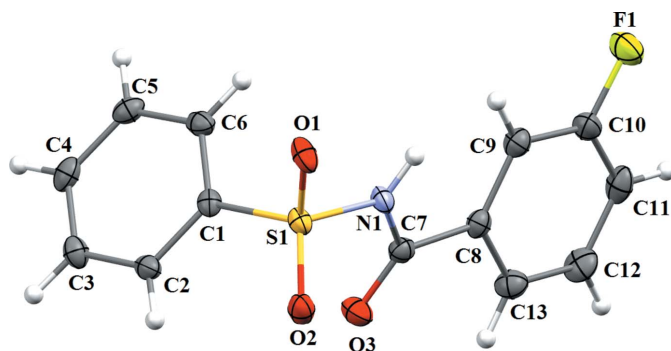


Figure 1
A view of the molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

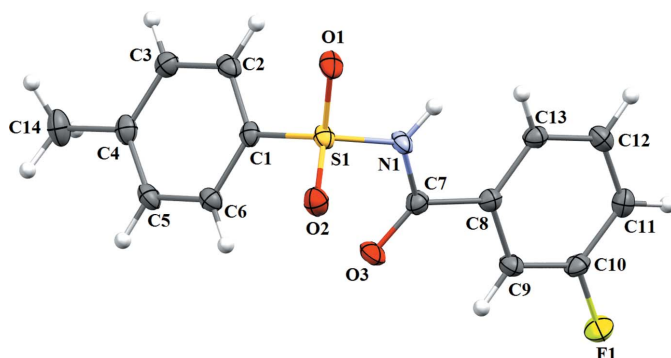


Figure 2
A view of the molecular structure of (II), with displacement ellipsoids drawn at the 50% probability level.

2. Structural commentary

The *meta*-fluoro substitution on the benzoyl ring of (I) (Fig. 1) is *syn* to the N–H bond in the central –C–SO₂–N–C(=O)– segment. By contrast, in (II) (Fig. 2), the conformation of the N–H bond is *anti* with respect to the *meta*-fluoro substitution on the benzoyl ring. The dihedral angle between the benzene rings is 82.73 (10)° in (I), while, in (II) the value is slightly less [72.60 (12)°]. Further, in (I), the dihedral angle between the benzoic acid ring and the central C8–C7(O3)–N1–S1 segment is 16.54 (10)°, while that between the sulfonamide ring and the C7(O3)–N1–S1–C1 segment is 81.87 (12)°. The corresponding values in (II) are slightly less than those observed in (I), being 12.12 (12) and 57.58 (13)°, respectively.

3. Supramolecular features

The crystal structure of (I) features strong N1–H1···O1 hydrogen bonds (Table 1) that connect the molecules into *R*₂²(8) dimers (Fig. 3). These dimers are further interconnected by C9–H9···O1 interactions, forming *R*₂²(14) ring motifs. C6–H6···O3 interactions connect these dimers into *C*₇ chains, forming columns propagating along the *b*-axis direction (Fig. 3). In addition, C4–H4···*C*_{g1} (π system of the

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

*C*_{g1} and *C*_{g2} are the centroids of the sulfonyl and benzoyl rings, respectively.

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1···O1 ⁱ	0.81 (3)	2.08 (3)	2.883 (2)	171 (3)
C9–H9···O1 ⁱ	0.93	2.42	3.244 (3)	148
C6–H6···O3 ⁱⁱ	0.93	2.50	3.294 (3)	143
C2–H2··· <i>C</i> _{g1} ⁱⁱⁱ	0.93	2.82	3.474 (2)	129
C4–H4··· <i>C</i> _{g2} ^{iv}	0.93	2.84	3.582 (2)	137
C11–H11··· <i>C</i> _{g1} ^v	0.93	2.97	3.756 (3)	143

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

fluorobenzoyl ring) interactions link the molecules into chains along the *c* axis. These chains are interconnected *via* C2–H2··· π _{aryl} (π system of the sulfonylbenzene ring) and C11–H11··· π _{aryl} (π system of the sulfonylbenzene ring) interactions, forming a three-dimensional grid-like structure (Fig. 4). The crystal structure also features π – π (π system of the fluorobenzoyl ring) stacking interactions. It is notable that the N–H···O hydrogen bonds present in the crystal structure of (I) has no structure-directing properties (leading only to dimers), while one of the C–H···O and the three C–H··· π _{aryl} interactions have structure-directing characteristics.

Similar to that observed in the crystal structure of (I), in (II) strong N1–H1···O1 hydrogen bonds (Table 2) result in the formation of *R*₂²(8) dimers (Fig. 5). The molecules constituting

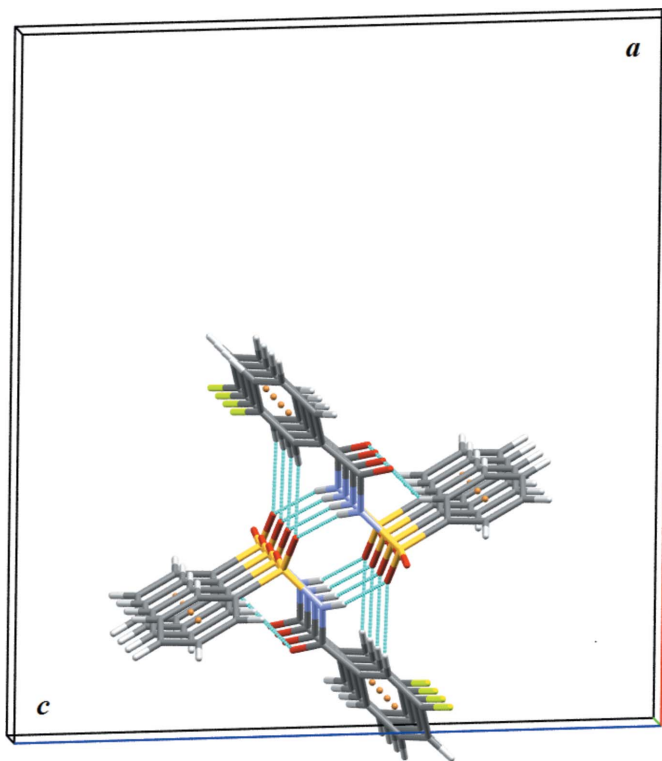


Figure 3
Crystal packing of (I), displaying N–H···O hydrogen bonds and C–H···O interactions, which result in columns along the *b* axis.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O1^i$	0.87 (4)	2.06 (4)	2.937 (3)	177 (3)
$C5-H5\cdots O3^{ii}$	0.93	2.46	3.375 (3)	168
$C13-H13\cdots O1^i$	0.93	2.47	3.285 (3)	147

Symmetry codes: (i) $-x + 2, -y + 1, -z + 2$; (ii) $-x + 1, -y + 1, -z + 1$.

these dimers are interconnected into $R_2^2(14)$ ring motifs *via* $C13-H13\cdots O1$ interactions, as observed in (I). Adjacent dimers are interconnected *via* $C5-H5\cdots O3$ interactions into $R_2^2(16)$ rings, thus forming ribbons along the diagonal of the ac plane (Fig. 5). The overall supramolecular architecture displayed in (II) is one-dimensional, in contrast to the three-dimensional architecture displayed in (I).

4. Database survey

The crystal structures of five related *N*-(arylsulfonyl)aryl-amides, namely *N*-(benzoyl)benzenesulfonamide (III), *N*-(3-chlorobenzoyl)benzenesulfonamide (IV), *N*-(3-methylbenzoyl)benzenesulfonamide (V), *N*-(benzoyl)-4-methylbenzenesulfonamide (VI) and *N*-(3-methylbenzoyl)-4-methylbenzenesulfonamide (VII) have previously been reported. A comparison of the dihedral angle between the two benzene rings in these closely related structures indicates that introducing a methyl substituent into the *para* position of the benzenesulfonyl ring lowers the dihedral angle with compound (VII) being an exception. The dihedral angle values are $80.3 (1)^\circ$ in (III) (Gowda *et al.*, 2009a), $87.5 (1)^\circ$ in (IV) (Gowda *et al.*, 2009b), $83.3 (2)$, $84.4 (2)$ and $87.6 (2)^\circ$ in

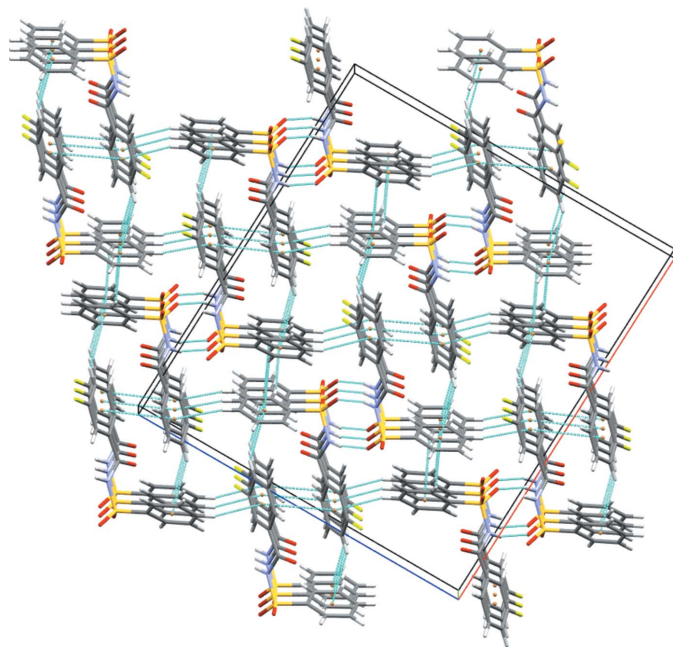


Figure 4
Three-dimensional grid-like architecture formed by various $C-H\cdots\pi_{\text{aryl}}$ interactions in (I).

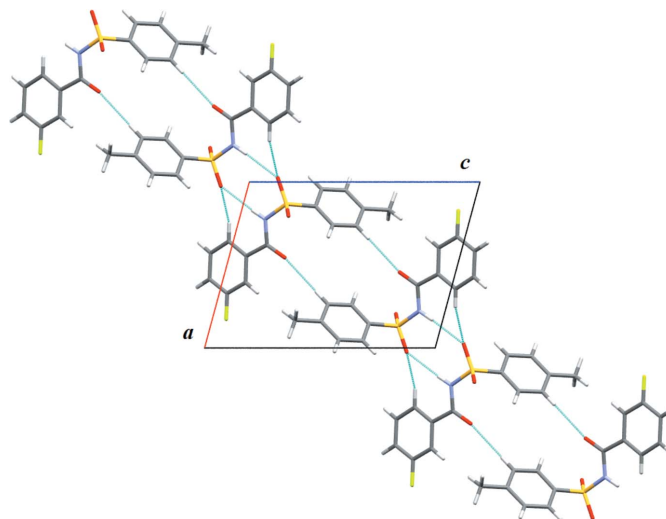


Figure 5
One-dimensional ribbons formed in the crystal structure of (II) *via* $N-H\cdots O$ dimeric pairs and various $C-H\cdots O$ dimeric pairs.

the three molecules of (V) (Suchetan *et al.*, 2012), $79.4 (1)^\circ$ in (VI) (Suchetan *et al.*, 2010) and $89.6 (2)^\circ$ in (VII) (Sreenivasa *et al.*, 2014). This effect is the same as that observed in the present two structures (I) and (II). Furthermore, in (I)–(VII) the conformation of the $N-H$ bond in the central segment is *anti* to the *meta* substituent on the benzoyl ring in the presence of a methyl substituent either on the benzoyl ring or the benzenesulfonyl ring. Otherwise, the conformation is *syn* as observed in (I) and (IV). A comparison of the crystal structures of (I) and (II) with those previously reported shows that fluoro substitution on the benzoyl ring appears to have a significant effect on the supramolecular architecture, and also on the type and nature of the intermolecular interactions displayed. For instance, in all the reported structures except (VII), the molecules are linked into one-dimensional infinite $C(4)$ chains *via* strong structure-directing $N-H\cdots O$ hydrogen bonds. The structures do not feature any other type of interactions. However, in (I) and (II), the $N-H\cdots O$ hydrogen bonds lead to dimers and, in addition, both of them feature other structure-directing interactions of the type $C-H\cdots O$ or $C-H\cdots\pi_{\text{aryl}}$. Furthermore, introducing the methyl substituent into the benzenesulfonyl ring of (I) to form (III) reduces the three-dimensional grid-like architecture into a one-dimensional ribbon architecture. However, in (III)–(VII), the introduction of a methyl substituent into the benzenesulfonyl ring results in no change to the supramolecular architecture.

5. Synthesis and crystallization

Compounds (I) and (II) were prepared by refluxing a mixture of 3-fluorobenzoic acid, the corresponding substituted benzenesulfonamides and phosphorus oxychloride for 3 h on a water bath. The resultant mixtures were cooled and poured into ice-cold water. The solids obtained were filtered, washed thoroughly with water and then dissolved in sodium bicarbo-

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₁₃ H ₁₀ FNO ₃ S	C ₁₄ H ₁₂ FNO ₃ S
<i>M_r</i>	279.28	293.31
Crystal system, space group	Monoclinic, <i>C2/c</i>	Monoclinic, <i>P2₁/c</i>
Temperature (K)	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	21.4036 (8), 5.7673 (2), 19.5525 (7)	9.0376 (4), 12.2912 (5), 12.1377 (5)
β (°)	92.135 (1)	105.107 (2)
<i>V</i> (Å ³)	2411.90 (15)	1301.70 (9)
<i>Z</i>	8	4
Radiation type	Cu <i>K</i> α	Cu <i>K</i> α
μ (mm ⁻¹)	2.56	2.40
Crystal size (mm)	0.28 × 0.24 × 0.19	0.28 × 0.22 × 0.18
Data collection		
Diffractometer	Bruker APEXII	Bruker APEXII
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2009)	Multi-scan (<i>SADABS</i> ; Bruker, 2009)
<i>T_{min}</i> , <i>T_{max}</i>	0.512, 0.614	0.557, 0.649
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	8647, 1985, 1846	8422, 2115, 1796
<i>R_{int}</i>	0.037	0.056
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.587	0.583
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.042, 0.133, 0.98	0.050, 0.152, 1.05
No. of reflections	1985	2115
No. of parameters	176	186
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.39, -0.38	0.45, -0.47

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

nate solutions. The compounds were later reprecipitated by acidifying the filtered solutions with dilute HCl. They were filtered, dried and recrystallized; m.p = 442–444 K for (I) and 422–423 K for (II). Prism-like, colourless single crystals of (I) and (II) were obtained by slow evaporation of the respective solutions of the compounds in methanol (with a few drops of water).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms of the NH groups in (I) and (II) were located in a difference map and later refined freely. The other H atoms were positioned with idealized geometry using a riding model with C–H = 0.93–0.96 Å, and with *U*_{iso} = 1.2 or 1.5*U*_{eq}(parent atom). To improve considerably the values of *R*1, *wR*2 and GOOF, reflections with very bad agreement (–20 0 0), (–20 0 10) and (–19 1 15) in (I) and (0 6 0) in (II) were omitted from the final refinements.

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. GMS thanks the Vision Group on Science and Technology (VGST), Karnataka, India, for financial support under its SPiCE project scheme.

References

- Banwell, M. G., Crasto, C. F., Easton, C. J., Forrest, A. K., Karoli, T., March, D. R., Mensah, L., Nairn, M. R., O'Hanlon, P. J., Oldham, M. D. & Yue, W. (2000). *Bioorg. Med. Chem. Lett.* **10**, 2263–2266.
- Bruker (2009). *APEX2, SADABS and SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chang, L. L., Ashton, W. T., Flanagan, K. L., Chen, T. B., O'Malley, S. S., Zingaro, G. J., Siegl, P. K. S., Kivlighn, S. D., Lotti, V. J., Chang, R. S. L. & Greenlee, W. J. (1994). *J. Med. Chem.* **37**, 4464–4478.
- Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2009a). *Acta Cryst.* **E65**, o2516.
- Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2009b). *Acta Cryst.* **E65**, o2750.
- Hasegawa, T. & Yamamoto, H. (2000). *Bull. Chem. Soc. Jpn.* **73**, 423–428.
- 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.
- Mader, M., Shih, C., Considine, E., Dios, A. D., Grossman, C., Hipskind, P., Lin, H., Lobb, K., Lopez, B., Lopez, J., Cabrejas, L., Richett, M., White, W., Cheung, Y., Huang, Z., Reilly, J. & Dinn, S. (2005). *Bioorg. Med. Chem. Lett.* **15**, 617–620.
- Musser, J. H., Kreft, A. F., Bender, R. H. W., Kubrak, D. M., Grimes, D., Carlson, R. P., Hand, J. M. & Chang, J. (1990). *J. Med. Chem.* **33**, 240–245.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sreenivasa, S., Mohan, N. R., Manojkumar, K. E. & Suchetan, P. A. (2014). *J. Appl. Chem.* **3**, 551–559.
- Suchetan, P. A., Foro, S. & Gowda, B. T. (2012). *Acta Cryst.* **E68**, o1327.
- Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010). *Acta Cryst.* **E66**, o1039.

supporting information

Acta Cryst. (2016). E72, 428–431 [https://doi.org/10.1107/S2056989016003248]

Crystal structures of *N*-(3-fluorobenzoyl)benzenesulfonamide and *N*-(3-fluorobenzoyl)-4-methylbenzenesulfonamide

P. A. Suchetan, S. Naveen, N. K. Lokanath, H. N. Lakshmikantha, K. S. Srivishnu and G. M. Supriya

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(I) *N*-(3-Fluorobenzoyl)benzenesulfonamide

Crystal data

$C_{13}H_{10}FNO_3S$

$M_r = 279.28$

Monoclinic, *C2/c*

Hall symbol: $-C\ 2yc$

$a = 21.4036\ (8)\ \text{\AA}$

$b = 5.7673\ (2)\ \text{\AA}$

$c = 19.5525\ (7)\ \text{\AA}$

$\beta = 92.135\ (1)^\circ$

$V = 2411.90\ (15)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1152$

Prism

$D_x = 1.538\ \text{Mg m}^{-3}$

Melting point: 442 K

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 123 reflections

$\theta = 4.1\text{--}64.8^\circ$

$\mu = 2.56\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Prism, colourless

$0.28 \times 0.24 \times 0.19\ \text{mm}$

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ϕ and ψ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

$T_{\min} = 0.512$, $T_{\max} = 0.614$

8647 measured reflections

1985 independent reflections

1846 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 64.8^\circ$, $\theta_{\min} = 4.1^\circ$

$h = -24 \rightarrow 24$

$k = -6 \rightarrow 5$

$l = -22 \rightarrow 22$

1 standard reflections every 1 reflections

intensity decay: 0.1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.133$

$S = 0.98$

1985 reflections

176 parameters

0 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

$$w = 1/[\sigma^2(F_o^2) + (0.105P)^2 + 2.9874P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.26504 (2)	0.47902 (9)	0.41244 (2)	0.0162 (2)
F1	0.46178 (7)	0.8164 (3)	0.66939 (7)	0.0325 (4)
O2	0.24567 (7)	0.2440 (3)	0.40478 (8)	0.0220 (4)
O1	0.21912 (7)	0.6490 (3)	0.42975 (8)	0.0230 (4)
O3	0.38206 (7)	0.2258 (3)	0.43259 (8)	0.0233 (4)
N1	0.31971 (9)	0.5057 (3)	0.47405 (10)	0.0174 (4)
C8	0.41781 (9)	0.4102 (4)	0.53566 (11)	0.0175 (5)
C7	0.37286 (10)	0.3690 (3)	0.47665 (11)	0.0176 (5)
C3	0.33096 (10)	0.4990 (4)	0.22344 (12)	0.0205 (5)
H3	0.3305	0.4048	0.1848	0.025*
C9	0.41783 (10)	0.6094 (4)	0.57623 (11)	0.0195 (5)
H9	0.3885	0.7264	0.5684	0.023*
C5	0.36070 (10)	0.8565 (4)	0.27931 (12)	0.0215 (5)
H5	0.3808	0.9994	0.2782	0.026*
C2	0.30217 (10)	0.4254 (4)	0.28191 (11)	0.0180 (5)
H2	0.2828	0.2811	0.2831	0.022*
C6	0.33094 (9)	0.7874 (4)	0.33818 (11)	0.0188 (5)
H6	0.3300	0.8840	0.3762	0.023*
C10	0.46279 (10)	0.6266 (4)	0.62832 (11)	0.0215 (5)
C1	0.30264 (9)	0.5700 (4)	0.33854 (10)	0.0153 (5)
C4	0.36042 (10)	0.7129 (4)	0.22245 (12)	0.0217 (5)
H4	0.3802	0.7605	0.1833	0.026*
C11	0.50814 (11)	0.4618 (4)	0.64123 (12)	0.0247 (5)
H11	0.5383	0.4820	0.6762	0.030*
C13	0.46262 (10)	0.2394 (4)	0.54849 (12)	0.0228 (5)
H13	0.4623	0.1061	0.5217	0.027*
C12	0.50767 (11)	0.2653 (4)	0.60072 (13)	0.0268 (6)
H12	0.5376	0.1504	0.6085	0.032*
H1	0.3091 (14)	0.591 (5)	0.5042 (17)	0.035 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0155 (4)	0.0201 (4)	0.0130 (3)	0.00064 (18)	-0.0003 (2)	-0.00239 (18)
F1	0.0361 (8)	0.0312 (8)	0.0295 (8)	-0.0004 (6)	-0.0089 (6)	-0.0093 (6)
O2	0.0238 (8)	0.0243 (9)	0.0178 (8)	-0.0059 (7)	-0.0004 (6)	0.0001 (6)
O1	0.0178 (8)	0.0339 (9)	0.0173 (8)	0.0065 (7)	-0.0013 (6)	-0.0070 (6)
O3	0.0233 (8)	0.0233 (8)	0.0233 (9)	0.0036 (6)	0.0000 (6)	-0.0069 (7)
N1	0.0185 (10)	0.0213 (10)	0.0124 (9)	0.0031 (7)	-0.0009 (8)	-0.0035 (7)
C8	0.0152 (10)	0.0203 (11)	0.0171 (11)	0.0004 (9)	0.0026 (8)	0.0027 (9)
C7	0.0187 (10)	0.0160 (11)	0.0183 (11)	-0.0003 (8)	0.0029 (8)	0.0014 (8)
C3	0.0194 (11)	0.0257 (12)	0.0165 (11)	0.0020 (8)	0.0009 (9)	-0.0024 (8)
C9	0.0170 (10)	0.0211 (11)	0.0205 (11)	0.0006 (8)	0.0015 (8)	0.0021 (9)
C5	0.0194 (11)	0.0164 (11)	0.0286 (12)	0.0014 (8)	-0.0020 (9)	0.0040 (9)
C2	0.0182 (11)	0.0167 (10)	0.0190 (11)	0.0022 (9)	-0.0024 (8)	-0.0022 (9)
C6	0.0183 (10)	0.0172 (11)	0.0208 (11)	0.0026 (8)	-0.0023 (8)	-0.0026 (9)
C10	0.0224 (11)	0.0218 (12)	0.0203 (11)	-0.0042 (9)	0.0008 (9)	-0.0006 (9)
C1	0.0132 (10)	0.0178 (10)	0.0149 (10)	0.0041 (8)	-0.0007 (8)	0.0006 (9)
C4	0.0166 (10)	0.0281 (12)	0.0207 (12)	0.0027 (9)	0.0018 (8)	0.0084 (9)
C11	0.0172 (11)	0.0336 (13)	0.0229 (12)	-0.0031 (9)	-0.0037 (9)	0.0070 (10)
C13	0.0193 (11)	0.0223 (11)	0.0268 (12)	0.0024 (9)	0.0020 (9)	0.0001 (9)
C12	0.0189 (11)	0.0288 (13)	0.0323 (13)	0.0066 (9)	-0.0032 (9)	0.0041 (10)

Geometric parameters (\AA , $^\circ$)

S1—O2	1.4238 (16)	C9—H9	0.9300
S1—O1	1.4375 (16)	C5—C4	1.386 (3)
S1—N1	1.6549 (19)	C5—C6	1.394 (3)
S1—C1	1.760 (2)	C5—H5	0.9300
F1—C10	1.358 (3)	C2—C1	1.386 (3)
O3—C7	1.215 (3)	C2—H2	0.9300
N1—C7	1.383 (3)	C6—C1	1.392 (3)
N1—H1	0.81 (3)	C6—H6	0.9300
C8—C13	1.391 (3)	C10—C11	1.375 (3)
C8—C9	1.396 (3)	C4—H4	0.9300
C8—C7	1.494 (3)	C11—C12	1.383 (4)
C3—C2	1.385 (3)	C11—H11	0.9300
C3—C4	1.386 (3)	C13—C12	1.386 (3)
C3—H3	0.9300	C13—H13	0.9300
C9—C10	1.378 (3)	C12—H12	0.9300
O2—S1—O1	118.36 (9)	C3—C2—C1	119.0 (2)
O2—S1—N1	111.12 (9)	C3—C2—H2	120.5
O1—S1—N1	103.67 (9)	C1—C2—H2	120.5
O2—S1—C1	109.75 (10)	C1—C6—C5	118.3 (2)
O1—S1—C1	109.12 (10)	C1—C6—H6	120.9
N1—S1—C1	103.73 (9)	C5—C6—H6	120.9
C7—N1—S1	122.13 (16)	F1—C10—C11	118.4 (2)

C7—N1—H1	125 (2)	F1—C10—C9	117.97 (19)
S1—N1—H1	112 (2)	C11—C10—C9	123.7 (2)
C13—C8—C9	119.6 (2)	C2—C1—C6	121.9 (2)
C13—C8—C7	116.5 (2)	C2—C1—S1	119.10 (17)
C9—C8—C7	123.80 (19)	C6—C1—S1	119.01 (16)
O3—C7—N1	121.08 (19)	C3—C4—C5	120.6 (2)
O3—C7—C8	122.62 (19)	C3—C4—H4	119.7
N1—C7—C8	116.29 (18)	C5—C4—H4	119.7
C2—C3—C4	120.1 (2)	C10—C11—C12	118.1 (2)
C2—C3—H3	120.0	C10—C11—H11	120.9
C4—C3—H3	120.0	C12—C11—H11	120.9
C10—C9—C8	117.7 (2)	C8—C13—C12	120.8 (2)
C10—C9—H9	121.2	C8—C13—H13	119.6
C8—C9—H9	121.2	C12—C13—H13	119.6
C4—C5—C6	120.2 (2)	C11—C12—C13	120.0 (2)
C4—C5—H5	119.9	C11—C12—H12	120.0
C6—C5—H5	119.9	C13—C12—H12	120.0
O2—S1—N1—C7	51.25 (19)	C5—C6—C1—C2	-1.5 (3)
O1—S1—N1—C7	179.42 (17)	C5—C6—C1—S1	179.79 (15)
C1—S1—N1—C7	-66.61 (19)	O2—S1—C1—C2	7.79 (19)
S1—N1—C7—O3	1.5 (3)	O1—S1—C1—C2	-123.41 (16)
S1—N1—C7—C8	-179.26 (14)	N1—S1—C1—C2	126.59 (16)
C13—C8—C7—O3	-16.3 (3)	O2—S1—C1—C6	-173.49 (15)
C9—C8—C7—O3	162.1 (2)	O1—S1—C1—C6	55.31 (18)
C13—C8—C7—N1	164.44 (19)	N1—S1—C1—C6	-54.69 (18)
C9—C8—C7—N1	-17.2 (3)	C2—C3—C4—C5	-0.9 (3)
C13—C8—C9—C10	-0.5 (3)	C6—C5—C4—C3	-0.3 (3)
C7—C8—C9—C10	-178.80 (19)	F1—C10—C11—C12	177.9 (2)
C4—C3—C2—C1	0.9 (3)	C9—C10—C11—C12	-1.6 (4)
C4—C5—C6—C1	1.5 (3)	C9—C8—C13—C12	-0.6 (3)
C8—C9—C10—F1	-177.86 (19)	C7—C8—C13—C12	177.9 (2)
C8—C9—C10—C11	1.6 (3)	C10—C11—C12—C13	0.5 (4)
C3—C2—C1—C6	0.3 (3)	C8—C13—C12—C11	0.6 (4)
C3—C2—C1—S1	179.00 (16)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg2 are the centroids of the sulfonyl and benzoyl rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1 ⁱ	0.81 (3)	2.08 (3)	2.883 (2)	171 (3)
C9—H9 \cdots O1 ⁱ	0.93	2.42	3.244 (3)	148
C6—H6 \cdots O3 ⁱⁱ	0.93	2.50	3.294 (3)	143
C2—H2 \cdots Cg1 ⁱⁱⁱ	0.93	2.82	3.474 (2)	129
C4—H4 \cdots Cg2 ^{iv}	0.93	2.84	3.582 (2)	137
C11—H11 \cdots Cg1 ^v	0.93	2.97	3.756 (3)	143

Symmetry codes: (i) $-x+1/2, -y+3/2, -z+1$; (ii) $x, y+1, z$; (iii) $x, -y-1, z-1/2$; (iv) $x, -y, z+1/2$; (v) $-x, -y, -z$.

(II) *N*-(3-Fluorobenzoyl)-4-methylbenzenesulfonamide*Crystal data*C₁₄H₁₂FNO₃S $M_r = 293.31$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 9.0376$ (4) Å $b = 12.2912$ (5) Å $c = 12.1377$ (5) Å $\beta = 105.107$ (2)° $V = 1301.70$ (9) Å³ $Z = 4$ $F(000) = 608$

Prism

 $D_x = 1.497$ Mg m⁻³

Melting point: 423 K

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 142 reflections

 $\theta = 5.1$ – 64.1 ° $\mu = 2.40$ mm⁻¹ $T = 173$ K

Prism, colourless

 $0.28 \times 0.22 \times 0.18$ mm*Data collection*

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ϕ and φ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.557$, $T_{\max} = 0.649$

8422 measured reflections

2115 independent reflections

1796 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.056$ $\theta_{\max} = 64.1$ °, $\theta_{\min} = 5.1$ ° $h = -10$ → 10 $k = -14$ → 14 $l = -10$ → 13

1 standard reflections every 1 reflections

intensity decay: 0.1%

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.152$ $S = 1.05$

2115 reflections

186 parameters

0 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

 $w = 1/[\sigma^2(F_o^2) + (0.1104P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.023$ $\Delta\rho_{\max} = 0.45$ e Å⁻³ $\Delta\rho_{\min} = -0.47$ 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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.86791 (7)	0.55562 (5)	0.81354 (5)	0.0171 (3)
F1	0.17463 (17)	0.30702 (14)	0.94022 (13)	0.0303 (4)

O1	1.0242 (2)	0.55041 (16)	0.88113 (15)	0.0231 (5)
O2	0.7956 (2)	0.65901 (15)	0.79047 (14)	0.0240 (5)
O3	0.5443 (2)	0.48889 (17)	0.75791 (15)	0.0260 (5)
N1	0.7768 (2)	0.47863 (18)	0.88728 (19)	0.0183 (5)
C8	0.5545 (3)	0.3863 (2)	0.9263 (2)	0.0169 (5)
C2	0.9568 (3)	0.4058 (2)	0.6775 (2)	0.0205 (6)
H2	1.0292	0.3827	0.7426	0.025*
C9	0.3950 (3)	0.3781 (2)	0.8985 (2)	0.0199 (6)
H9	0.3346	0.4151	0.8360	0.024*
C13	0.6429 (3)	0.3294 (2)	1.0197 (2)	0.0192 (6)
H13	0.7492	0.3351	1.0387	0.023*
C12	0.5722 (3)	0.2640 (2)	1.0844 (2)	0.0206 (6)
H12	0.6318	0.2251	1.1457	0.025*
C5	0.7362 (3)	0.4729 (2)	0.4840 (2)	0.0214 (6)
H5	0.6619	0.4947	0.4193	0.026*
C3	0.9501 (3)	0.3593 (2)	0.5727 (2)	0.0204 (6)
H3	1.0200	0.3053	0.5672	0.024*
C10	0.3296 (3)	0.3141 (2)	0.9655 (2)	0.0207 (6)
C4	0.8399 (3)	0.3922 (2)	0.4747 (2)	0.0210 (6)
C11	0.4148 (3)	0.2562 (2)	1.0587 (2)	0.0212 (6)
H11	0.3671	0.2134	1.1026	0.025*
C6	0.7415 (3)	0.5214 (2)	0.5878 (2)	0.0189 (6)
H6	0.6718	0.5754	0.5931	0.023*
C7	0.6213 (3)	0.4561 (2)	0.8499 (2)	0.0191 (6)
C1	0.8532 (3)	0.4879 (2)	0.6840 (2)	0.0169 (6)
C14	0.8314 (4)	0.3399 (2)	0.3606 (2)	0.0299 (7)
H14A	0.7802	0.2710	0.3563	0.045*
H14B	0.9331	0.3291	0.3524	0.045*
H14C	0.7754	0.3864	0.3006	0.045*
H1	0.833 (4)	0.469 (3)	0.957 (3)	0.033 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0170 (4)	0.0203 (4)	0.0131 (4)	−0.0036 (2)	0.0024 (3)	−0.0003 (2)
F1	0.0154 (8)	0.0438 (11)	0.0311 (9)	−0.0039 (7)	0.0050 (6)	0.0043 (7)
O1	0.0181 (10)	0.0340 (12)	0.0153 (9)	−0.0082 (8)	0.0011 (7)	−0.0010 (7)
O2	0.0298 (10)	0.0223 (10)	0.0204 (10)	−0.0003 (8)	0.0075 (8)	−0.0008 (7)
O3	0.0173 (10)	0.0386 (12)	0.0184 (10)	−0.0005 (8)	−0.0019 (8)	0.0092 (8)
N1	0.0152 (11)	0.0245 (12)	0.0127 (11)	−0.0033 (9)	−0.0006 (9)	0.0027 (9)
C8	0.0159 (12)	0.0170 (13)	0.0172 (12)	−0.0003 (10)	0.0032 (9)	−0.0031 (10)
C2	0.0161 (12)	0.0251 (15)	0.0181 (13)	−0.0024 (11)	0.0004 (10)	0.0029 (10)
C9	0.0175 (12)	0.0233 (14)	0.0169 (13)	0.0008 (11)	0.0012 (10)	0.0015 (10)
C13	0.0164 (12)	0.0210 (14)	0.0192 (13)	0.0010 (10)	0.0030 (10)	−0.0021 (10)
C12	0.0250 (13)	0.0183 (14)	0.0167 (12)	0.0011 (11)	0.0020 (10)	0.0015 (10)
C5	0.0216 (13)	0.0255 (14)	0.0141 (13)	−0.0027 (11)	−0.0005 (10)	0.0027 (10)
C3	0.0204 (13)	0.0188 (13)	0.0227 (13)	−0.0006 (11)	0.0070 (10)	0.0005 (10)
C10	0.0128 (12)	0.0273 (15)	0.0215 (13)	−0.0009 (10)	0.0035 (10)	−0.0044 (10)

C4	0.0242 (14)	0.0212 (14)	0.0186 (13)	-0.0070 (11)	0.0074 (10)	-0.0004 (10)
C11	0.0252 (13)	0.0207 (14)	0.0201 (13)	-0.0025 (11)	0.0101 (11)	-0.0010 (10)
C6	0.0183 (13)	0.0199 (13)	0.0170 (13)	-0.0014 (11)	0.0020 (10)	0.0008 (10)
C7	0.0176 (13)	0.0224 (15)	0.0173 (13)	-0.0005 (11)	0.0048 (11)	-0.0018 (10)
C1	0.0165 (13)	0.0187 (13)	0.0161 (13)	-0.0044 (10)	0.0054 (10)	0.0001 (10)
C14	0.0428 (17)	0.0295 (16)	0.0189 (13)	-0.0069 (13)	0.0103 (12)	-0.0024 (11)

Geometric parameters (Å, °)

S1—O2	1.423 (2)	C13—H13	0.9300
S1—O1	1.4383 (19)	C12—C11	1.378 (4)
S1—N1	1.661 (2)	C12—H12	0.9300
S1—C1	1.753 (2)	C5—C6	1.383 (4)
F1—C10	1.356 (3)	C5—C4	1.389 (4)
O3—C7	1.220 (3)	C5—H5	0.9300
N1—C7	1.387 (3)	C3—C4	1.398 (4)
N1—H1	0.87 (4)	C3—H3	0.9300
C8—C13	1.393 (4)	C10—C11	1.388 (4)
C8—C9	1.396 (4)	C4—C14	1.510 (4)
C8—C7	1.500 (4)	C11—H11	0.9300
C2—C3	1.382 (4)	C6—C1	1.392 (4)
C2—C1	1.393 (4)	C6—H6	0.9300
C2—H2	0.9300	C14—H14A	0.9600
C9—C10	1.371 (4)	C14—H14B	0.9600
C9—H9	0.9300	C14—H14C	0.9600
C13—C12	1.391 (4)		
O2—S1—O1	118.99 (11)	C2—C3—C4	120.9 (2)
O2—S1—N1	110.36 (11)	C2—C3—H3	119.5
O1—S1—N1	102.63 (11)	C4—C3—H3	119.5
O2—S1—C1	108.91 (11)	F1—C10—C9	118.8 (2)
O1—S1—C1	108.91 (11)	F1—C10—C11	118.2 (2)
N1—S1—C1	106.26 (11)	C9—C10—C11	123.0 (2)
C7—N1—S1	122.65 (19)	C5—C4—C3	118.9 (2)
C7—N1—H1	125 (2)	C5—C4—C14	120.3 (2)
S1—N1—H1	111 (2)	C3—C4—C14	120.8 (2)
C13—C8—C9	119.8 (2)	C12—C11—C10	118.0 (2)
C13—C8—C7	123.5 (2)	C12—C11—H11	121.0
C9—C8—C7	116.7 (2)	C10—C11—H11	121.0
C3—C2—C1	118.9 (2)	C5—C6—C1	118.9 (2)
C3—C2—H2	120.5	C5—C6—H6	120.6
C1—C2—H2	120.5	C1—C6—H6	120.6
C10—C9—C8	118.5 (2)	O3—C7—N1	121.4 (2)
C10—C9—H9	120.8	O3—C7—C8	121.9 (2)
C8—C9—H9	120.8	N1—C7—C8	116.6 (2)
C12—C13—C8	119.9 (2)	C6—C1—C2	121.1 (2)
C12—C13—H13	120.0	C6—C1—S1	118.8 (2)
C8—C13—H13	120.0	C2—C1—S1	120.01 (19)

C11—C12—C13	120.8 (2)	C4—C14—H14A	109.5
C11—C12—H12	119.6	C4—C14—H14B	109.5
C13—C12—H12	119.6	H14A—C14—H14B	109.5
C6—C5—C4	121.2 (2)	C4—C14—H14C	109.5
C6—C5—H5	119.4	H14A—C14—H14C	109.5
C4—C5—H5	119.4	H14B—C14—H14C	109.5
O2—S1—N1—C7	55.4 (2)	C4—C5—C6—C1	-0.1 (4)
O1—S1—N1—C7	-176.8 (2)	S1—N1—C7—O3	2.7 (4)
C1—S1—N1—C7	-62.5 (2)	S1—N1—C7—C8	-179.31 (17)
C13—C8—C9—C10	0.6 (4)	C13—C8—C7—O3	166.5 (3)
C7—C8—C9—C10	179.3 (2)	C9—C8—C7—O3	-12.1 (4)
C9—C8—C13—C12	0.4 (4)	C13—C8—C7—N1	-11.5 (4)
C7—C8—C13—C12	-178.2 (2)	C9—C8—C7—N1	169.9 (2)
C8—C13—C12—C11	-1.3 (4)	C5—C6—C1—C2	-1.1 (4)
C1—C2—C3—C4	-1.2 (4)	C5—C6—C1—S1	175.79 (19)
C8—C9—C10—F1	179.0 (2)	C3—C2—C1—C6	1.7 (4)
C8—C9—C10—C11	-0.9 (4)	C3—C2—C1—S1	-175.14 (18)
C6—C5—C4—C3	0.6 (4)	O2—S1—C1—C6	-19.8 (2)
C6—C5—C4—C14	179.7 (2)	O1—S1—C1—C6	-150.97 (19)
C2—C3—C4—C5	0.0 (4)	N1—S1—C1—C6	99.1 (2)
C2—C3—C4—C14	-179.0 (2)	O2—S1—C1—C2	157.1 (2)
C13—C12—C11—C10	1.0 (4)	O1—S1—C1—C2	25.9 (2)
F1—C10—C11—C12	-179.7 (2)	N1—S1—C1—C2	-84.0 (2)
C9—C10—C11—C12	0.1 (4)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1 ⁱ	0.87 (4)	2.06 (4)	2.937 (3)	177 (3)
C5—H5 \cdots O3 ⁱⁱ	0.93	2.46	3.375 (3)	168
C13—H13 \cdots O1 ⁱ	0.93	2.47	3.285 (3)	147

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $-x+1, -y+1, -z+1$.