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Crystal structures of three *N*-(arylsulfonyl)-4-fluorobenzamides

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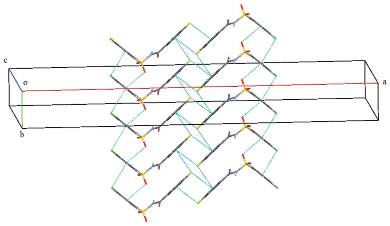
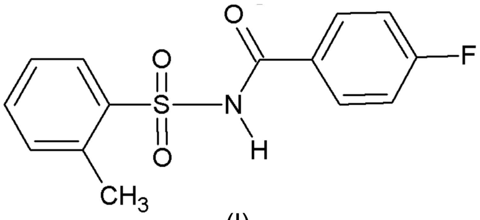
The crystal structures of three *N*-arylsulfonyl-4-fluorobenzamides, namely 4-fluoro-*N*-(2-methylphenylsulfonyl)benzamide, $C_{14}H_{12}FNO_3S$, (I), *N*-(2-chlorophenylsulfonyl)-4-fluorobenzamide, $C_{13}H_9ClFNO_3S$, (II), and *N*-(4-chlorophenylsulfonyl)-4-fluorobenzamide monohydrate, $C_{13}H_9ClFNO_3S\cdot H_2O$, (III), are described and compared with related structures. The asymmetric unit of (I) contains two independent molecules (*A* and *B*), while that of (II) contains just one molecule, and that of (III) contains a molecule of water in addition to one main molecule. The dihedral angle between the benzene rings is 82.83 (11)° in molecule *A* and 85.01 (10)° in molecule *B* of (I), compared to 89.91 (10)° in (II) and 81.82 (11)° in (III). The crystal structure of (I) features strong N—H···O hydrogen bonds between the *A* and *B* molecules, resulting in an $R_4^4(16)$ tetrameric unit. These tetrameric units are connected into sheets in the *bc* plane by various C—H···O interactions, and adjacent sheets are further interlinked *via* C—H···π_{aryl} interactions, forming a three-dimensional architecture. The crystal structure is further stabilized by π_{aryl}–π_{aryl} and S═O···π_{aryl} interactions. In the crystal of (II), molecules are connected into $R_2^2(8)$ and $R_2^2(14)$ dimers *via* N—H···O hydrogen bonds and C—H···O interactions, respectively; the dimers are further interconnected *via* a weak C═O···π_{aryl} interaction, leading to the formation of chains along $[1\bar{1}0]$. In the crystal of (III), N—H···O and O—H···O hydrogen bonds involving both the main molecule and the solvent water molecule results in the formation of sheets parallel to the *bc* plane. The sheets are further connected by C—H···O interactions and weak C—Cl···π_{aryl}, C—F···π_{aryl} and S═O···π_{aryl} interactions, forming a three-dimensional architecture.

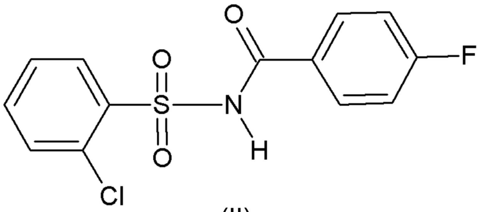
1. Chemical context

Sulfonamide and amide moieties play a very significant role as key constituents in a number of biologically active molecules (Mohan *et al.*, 2013; Manojkumar *et al.*, 2013; Hamad & Abed, 2014). In recent years, *N*-(arylsulfonyl)arylamides have received much attention as they constitute an important class of drugs for Alzheimer's disease (Hasegawa & Yamamoto, 2000), as well as antibacterial inhibitors of tRNA synthetases (Banwell *et al.*, 2000), antagonists for angiotensin II (Chang *et al.*, 1994) and leukotriene D4-receptors (Musser *et al.*, 1990). Further, *N*-(arylsulfonyl)arylamides are known to be potent anti-tumour agents against a broad spectrum of human tumour xenografts (colon, lung, breast, ovary and prostate) in nude mice (Mader *et al.*, 2005). In view of the importance of *N*-(arylsulfonyl)arylamides, the title compounds, (I), (II) and (III), were synthesized and we report herein on their crystal structures.

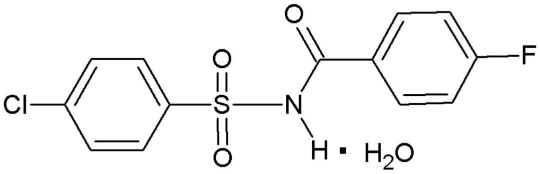




(I)



(II)



(III)

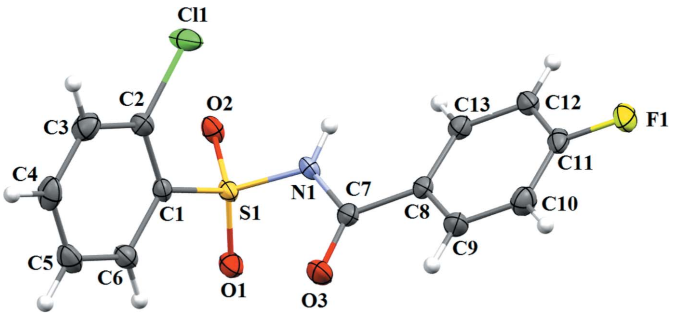


Figure 2
A view of the molecular structure of compound (II), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

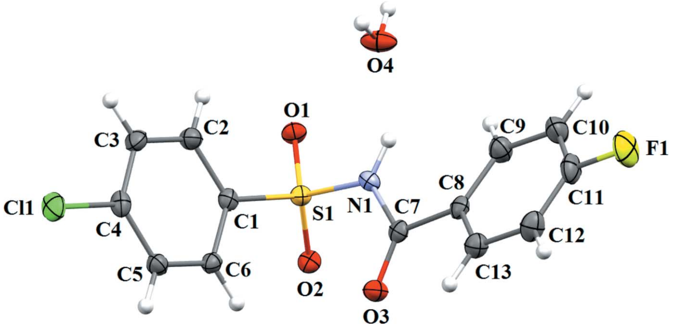


Figure 3
A view of the molecular structure of compound (III), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

The asymmetric unit of compound (I) contains two independent molecules (*A* and *B*) (Fig. 1), that differ slightly in their molecular conformations. The asymmetric unit of compound (II) (Fig. 2) contains one molecule, while compound (III) (Fig. 3) crystallizes as a water monosolvate. In molecules *A* and *B* of (I), the *ortho*-methyl substituent on the benzenesulfonyl ring is *syn* to the N—H bond in the central –C–SO_2–N–C(O)– segment (Fig. 1). This is similar to the *syn* conformation observed for the N—H bond in the central –C–SO_2–N–C(O)– segment with respect to the *ortho*-chloro substitution on the benzenesulfonyl ring of (II). The dihedral angle between the benzene rings is 82.83 (11)° in molecule *A* and 85.01 (10)° in molecule *B* of (I), compared to 89.91 (10)° in (II) and 81.82 (11)° in (III). Further, in (I) the dihedral angles between the benzoic acid ring and the central C8–C7(O3)–N1–S1 segment are 28.99 (1) and 23.81 (9)° in molecules *A* and *B*, respectively, while it is 10.41 (10)° in (II) and 21.23 (10)° in (III). The dihedral angles between the sulfonamide ring and the C7(O3)–N1–S1–C1 segment are, respectively, 68.67 (12) and 77.31 (10)° in molecules *A* and *B* of (I). The corresponding dihedral angle in (II) is 70.77 (11)°, whereas in (III) the value is much less, *viz* 48.03 (12)°. An intramolecular C14*B*--H14*B*···O2*B* hydrogen bond (Fig.1 and Table 1) is observed in molecule *B* of (I), with an *S*(6) ring motif.

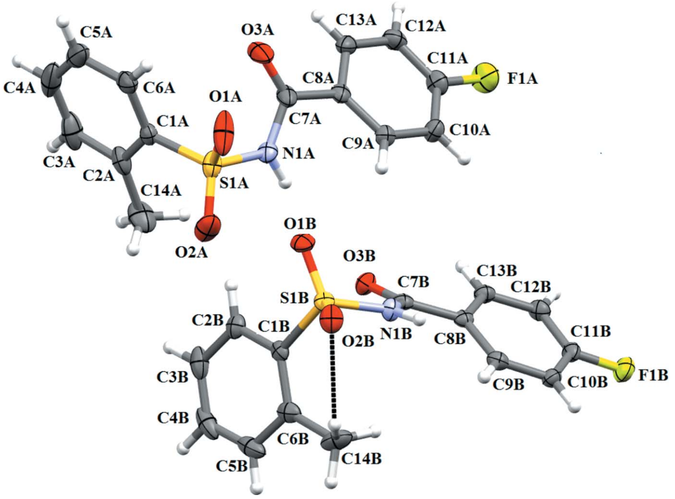


Figure 1
A view of the molecular structure of the two independent molecules (*A* and *B*) of compound (I), with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

3. Supramolecular features

The crystal structure of (I), features two strong N—H···O hydrogen bonds, namely, N1*A*—H1*A*···O1*B* and N1*B*—H1*B*···O1*A* hydrogen bonds (Table 1) between the *A* and *B* molecules, resulting in a tetrameric unit (Fig. 4). The unitary level graph-set notation for each hydrogen bond is *D*(2), while in the second level the tetrameric unit has a graph-set motif of $R_4^4(16)$. Adjacent tetramers are connected into sheets in the *bc* plane (Fig. 4) *via* C6*A*—H6*A*···O3*B*, C10*B*—H10*B*···O1*B*

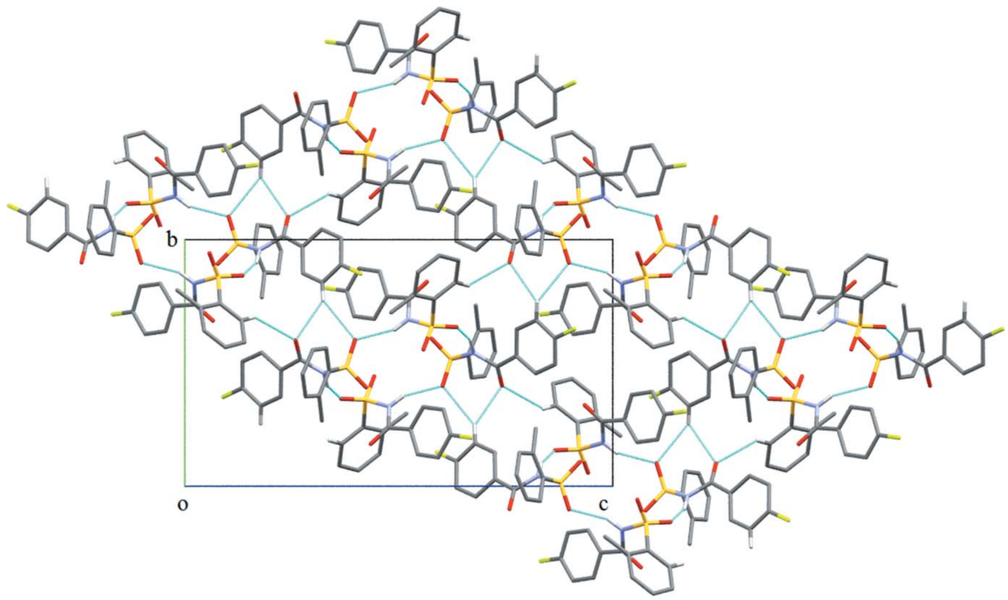


Figure 4
Crystal packing of (I), displaying N—H···O hydrogen bonds and C—H···O intermolecular interactions (dashed lines), which result in the formation of sheets parallel to the *bc* plane.

and C10*B*—H10*B*···O3*B* interactions (Table 1). Adjacent sheets are further interconnected *via* C4*B*—H4*B*···$\pi_{\rm aryl}$ interactions (involving the centroid of the fluorobenzoyl ring of molecule *B*) (Fig. 5 and Table 1) to form chains along the *a* axis, so forming a three-dimensional architecture. The crystal structure of (I), is further stabilized by $\pi_{\rm aryl}$–$\pi_{\rm aryl}$ interactions (Fig. 6) [$Cg1\cdots Cg2$ = 3.7413 (12) Å; *Cg*1 and *Cg*2 are the centroids of the fluorobenzoyl rings of molecules *A* and *B*, respectively] and also by weak S1*A*=O2*A*···$\pi_{\rm aryl}$ interactions [O···*Cg*3 = 3.7991 (19) Å; *Cg*3 is the centroid of the benzenesulfonyl ring of molecule *B*].

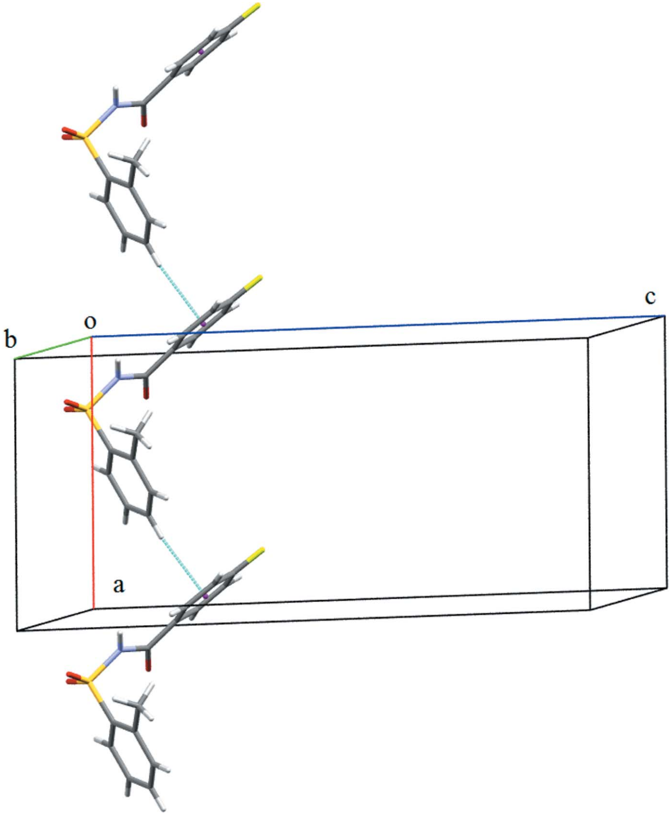


Figure 5
C—H···$\pi_{\rm aryl}$ interactions (dashed lines) displayed in (I).

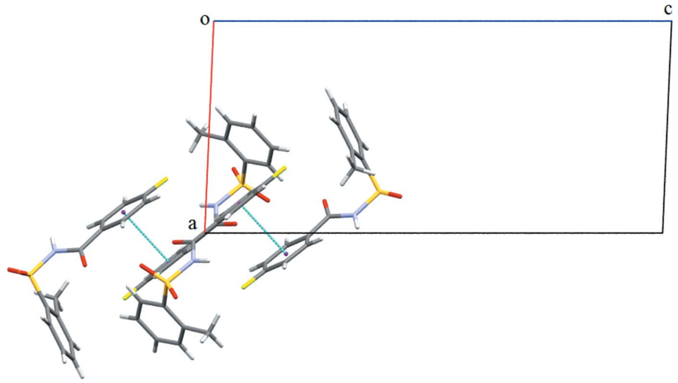


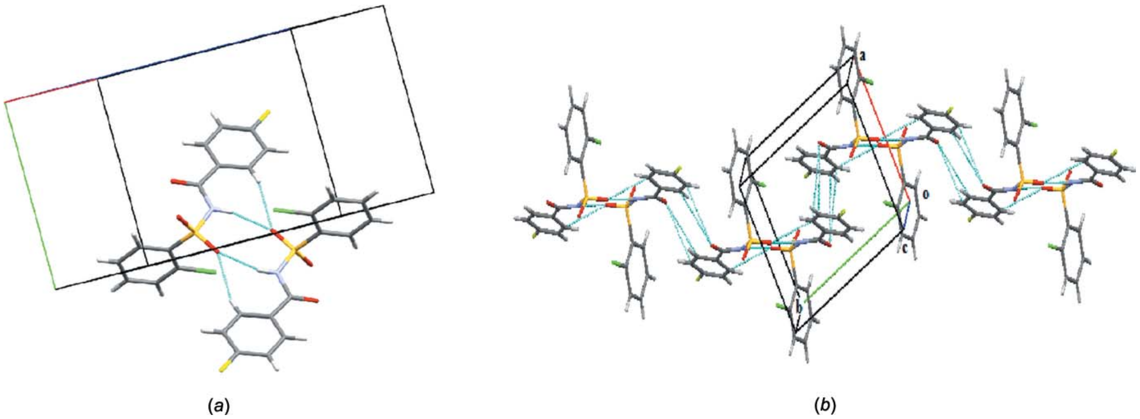
Figure 6
π–π interactions (dashed lines) displayed in (I).

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

Cg is the centroid of the fluorobenzene ring of molecule *B* of (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*B*	0.81 (3)	2.12 (3)	2.918 (2)	167 (2)
N1*B*—H1*B*···O1*A*$^{\rm i}$	0.83 (3)	2.02 (3)	2.828 (3)	162 (3)
C6*A*—H6*A*···O3*B*	0.93	2.57	3.313 (3)	137
C10*B*—H10*B*···O1*B*$^{\rm ii}$	0.93	2.59	3.376 (2)	143
C10*B*—H10*B*···O3*B*$^{\rm ii}$	0.93	2.46	3.215 (2)	139
C4*B*—H4*B*···*Cg*$^{\rm iii}$	0.93	2.72	3.646 (2)	173
C14*B*—H14*B*···O2*B*	0.96	2.45	3.058 (3)	121

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



(a) (b)

Figure 7
Crystal packing of (II): (*a*)display of $R_2^2(8)$ and $R_2^2(14)$ dimers formed *via* N—H⋯O hydrogen bonds and C—H⋯O interactions, both shown as dashed lines; (*b*) formation of one-dimensional architecture.

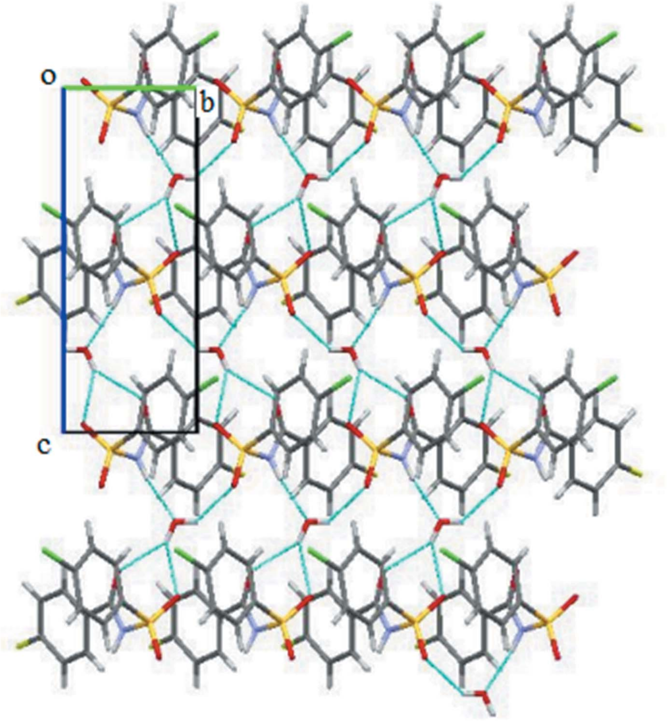


Figure 8
Crystal packing of (III), displaying an infinite two-dimensional sheet parallel to the *bc* plane formed *via* N—H⋯O and various O—H⋯O hydrogen bonds (dashed lines).

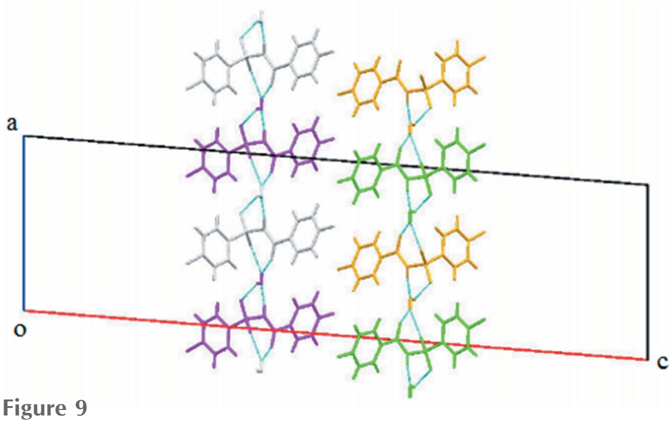


Figure 9
Crystal packing of (III) when viewed along the *b* axis; adjacent two-dimensional sheets are seen.

In the crystal of (II), molecules are connected into $R_2^2(8)$ dimers *via* N1—H1⋯O2 hydrogen bonds (Fig. 7*a* and Table 2), and the dimers are further interconnected *via* C13—H13⋯O2 interactions (Fig. 7*a* and Table 2) with an $R_2^2(14)$ graph-set motif. Weak C7═O3⋯$\pi_{\rm aryl}$ interactions [O⋯*Cg* = 3.9157 (19) Å; *Cg* is the centroid of the fluorobenzoyl ring] connect these dimers, thus forming a one-dimensional architecture (Fig. 7*b*).

In the crystal of (III), molecules are connected *via* bridging water molecules, through strong N1—H1⋯O4, O4—H1*O*4⋯O1, O4—H2*O*4⋯O2 and O4—H2*O*4⋯O3 hydrogen bonds (Table 3), resulting in the formation of sheets parallel to the *bc* plane (Figs. 8 and 9). The sheets are further connected by C5—H5⋯O1 interactions, forming *C*6 chains (Table 3) running parallel to the *c* axis (Fig. 10). The crystal structure is also stabilized by several weak C—H⋯π interactions, C4—Cl1⋯*Cg*1 [Cl⋯*Cg*1 = 3.7513 (11) Å], C11—F1⋯*Cg*2 [F1⋯*Cg*2 = 3.8674 (17) Å] and S1═O2⋯*Cg*1 interactions [O2⋯*Cg*1 = 3.2039 (17) Å] (*Cg*1 and *Cg*2 are the centroids of the benzenesulfonyl ring and fluorobenzoyl rings, respectively), forming a complex three-dimensional architecture (Fig. 11).

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯O2$^{\rm i}$	0.82 (3)	2.16 (3)	2.968 (2)	172 (2)
C13—H13⋯O2$^{\rm i}$	0.93	2.40	3.194 (2)	144

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯O4	0.83 (3)	1.91 (3)	2.733 (3)	171 (2)
O4—H1*O*4⋯O1$^{\rm i}$	0.79 (3)	2.25 (3)	2.884 (2)	138 (3)
O4—H2*O*4⋯O2$^{\rm ii}$	0.82 (2)	2.29 (3)	2.955 (2)	139 (3)
O4—H2*O*4⋯O3$^{\rm ii}$	0.82 (2)	2.16 (3)	2.841 (2)	141 (3)
C5—H5⋯O1$^{\rm iii}$	0.93	2.54	3.124 (3)	121

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

Table 4
Comparison of various parameters (°) in the crystal structures of series 1: *N*-(2-methylphenylsulfonyl)-*para*-substituted-arylamides.

Parameters	H	Cl	CH_3	NO_2	OCH_3	F
Crystal System	Orthorhombic	Triclinic	Triclinic	Monoclinic	Monoclinic	Monoclinic
Z'	1	2	2	1	1	2
Orientation of 2-CH_3 group to the N—H bond	*syn*	*syn*, *syn*	*syn*, *syn*	*syn*	*syn*	*syn*, *syn*
Angle between aromatic rings	73.9 (1)	89.4 (1), 82.4 (1)	88.1 (1), 83.5 (1)	83.8 (2)	80.81 (1)	82.83 (11), 85.01 (10)
Intermolecular interactions	N—H···O(S)	N—H···O(S)	N—H···O(S)	N—H···O(S)	N—H···O(S), C—H···O(S), π–π	N—H···O(S), C—H···O(S), C—H···π, π–π, S═O···π
Supramolecular architecture	0D chains	0D	0D chains	0D chains	1D chains	3D

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.37, last update February 2016; Groom & Allen, 2014) for similar compounds *viz* *N*-(arylsulfonyl)-4-(*substitusubstituted*)benzamides, gave 14 hits. These fourteen compounds along with the three title compounds, (I)–(III), are grouped into three series; **series 1**: *N*-(2-methylphenylsulfonyl)benzamide, *N*-(2-methylphenylsulfonyl)-4-(*chloro/methyl/nitro/methoxy*)benzamides and (I), **series 2**: *N*-(2-chlorophenylsulfonyl)benzamide, *N*-(2-chlorophenylsulfonyl)-4-(*chloro/methyl/nitro/methoxy*)benzamides and (II), and **series 3**: *N*-(4-chlorophenylsulfonyl)benzamide, *N*-(4-chlorophenylsulfonyl)-4-(*chloro/methyl/nitro*)benzamides and (III).

Series 1: In series 1 (Table 4), the asymmetric units of three compounds, namely, *N*-(2-methylphenylsulfonyl)benzamide (Suchetan *et al.*, 2010*d*), *N*-(2-methylphenylsulfonyl)-4-nitrobenzamide (Suchetan *et al.*, 2011*b*) and *N*-(2-methylphenylsulfonyl)-4-methoxybenzamide (Sreenivasa *et al.*, 2014*a*) contain one molecule, while those of *N*-(2-methylphenylsulfonyl)-4-chlorobenzamide (Suchetan *et al.*, 2010*e*), *N*-(2-methylphenylsulfonyl)-4-methylbenzamide (Gowda *et al.*, 2010*a*) and *N*-(2-methylphenylsulfonyl)-4-fluorobenzamide (I) contain two molecules. In all of the compounds of series 1, the conformation of the *ortho*-methyl group on the benzenesulfonyl ring is *syn* to the N—H bond in the central –C–SO_2-N–C(O)– segment. The values of the dihedral angle between the two aromatic rings in the molecules of series 1 fall in the range 73.9 (1)– 89.4 (1)°, the smallest dihedral angle being in *N*-(2-methylphenylsulfonyl)benzamide and the largest in *N*-(2-methylphenylsulfonyl)-4-chlorobenzamide (Table 4). Comparison of the intermolecular interactions displayed in the crystal structures of compounds in this series reveals that, except for the methoxy- and fluoro-substituted compounds, the crystal structures all display N—H···O(S) hydrogen bonds, while the methoxy- and fluoro-substituted compounds display other weak interactions of the type C—H···O, C—H···π_{aryl}, π_{aryl}–π_{aryl} in addition to the N—H···O(S) hydrogen bonds. However, except for compound (I), all the compounds display one-dimensional supramolecular chains, whereas in (I), the supramolecular architecture is three-dimensional.

Series 2: The asymmetric units of all of the compounds in series 2 (Table 5) contain one molecule and the conformation of the *ortho*-chloro substituent on the benzenesulfonyl ring is *syn* to the N—H bond in the central –C–SO_2–N–C(O)– segment. The values of the dihedral angle between the two

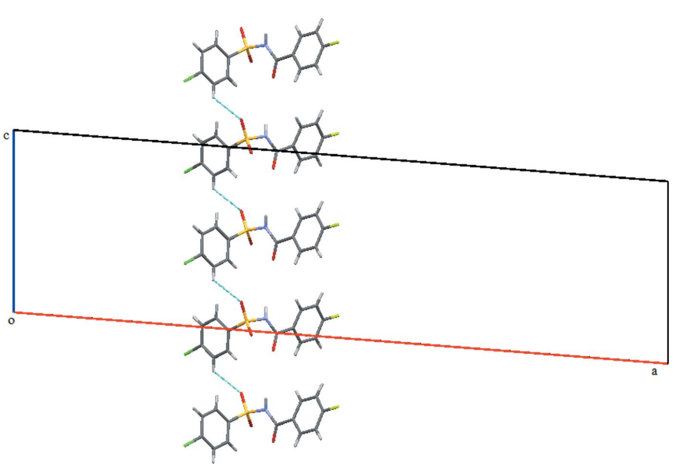


Figure 10
Display of C5—H5···O1 *C*(6) chains (dashed lines) running parallel to the *c* axis in (III).

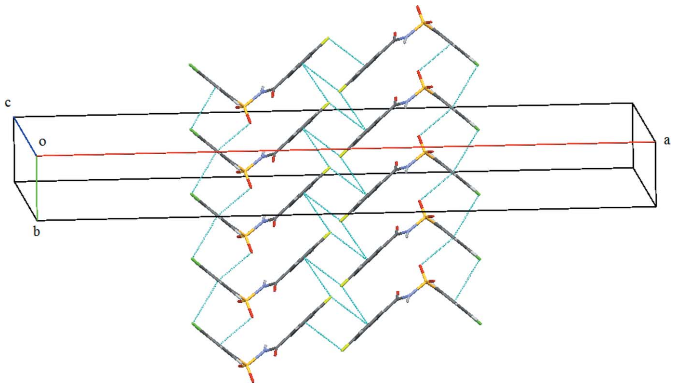


Figure 11
Display of various weak interactions (dashed lines) in the crystal structure of (III).

Table 5
Comparison of various parameters (°) in the crystal structures of series 2: *N*-(2-chlorophenylsulfonyl)-*para*-substituted-arylamides.

Parameters	H	Cl	CH_3	NO_2	OCH_3	F
Crystal System	Triclinic	Triclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Z′	1	1	1	1	1	1
Orientation of 2-Cl group to the N—H bond	*syn*	*syn*	*syn*	*syn*	*syn*	*syn*
Angle between aromatic rings	73.3 (1)	85.7 (1)	89.1 (2)	85.4 (1)	82.07 (1)	89.9 (1)
Intermolecular interactions	N—H···O(S)	N—H···O(S)	N—H···O(S)	N—H···O(S)	N—H···O(S), C—H···O(S), π–p	N—H···O(S), C—H···O(S), C═O···π
Supramolecular architecture	0D (ring motifs)	0D (ring motifs)	0D (ring motifs)	1D chains	2D	1D

Table 6
Comparison of various parameters (°) in the crystal structures of series 3: *N*-(4-chlorophenylsulfonyl)-*para*-substituted-arylamides.

Parameters	H	Cl	CH_3	NO_2	F
Crystal System	Triclinic	Orthorhombic	Orthorhombic	Monoclinic	Monoclinic
Z′	2	1	1	1	1, H_2O
Angle between aromatic rings	62.8 (1), 78.6 (1)	85.6 (1)	89.5 (1)	87.8 (1)	81.82 (11)
Intermolecular interactions	N—H···O(S)	N—H···O(S)	N—H···O(C)	N—H···O(S)	N—H···O(*W*), O(*W*)—H···O(S), O(*W*)—H···O(C), C—H···O(S), C—Cl···π, C—F···π, S═O···π
Supramolecular architecture	0D (ring motifs)	0D chains	0D (ring motifs)	D chains	3D

aromatic rings in the molecules fall in the range 73.3 (1)–89.91 (10)°, which is almost the same as in series 1, the smallest being in *N*-(2-chlorophenylsulfonyl)benzamide (Gowda *et al.*, 2010*b*) and the largest in *N*-(2-chlorophenylsulfonyl)-4-fluorobenzamide (II) (Table 5). The crystal structures of *N*-(2-chlorophenylsulfonyl)-benzamide, *N*-(2-chlorophenylsulfonyl)-4-chlorobenzamide (Suchetan *et al.*, 2011*c*) and *N*-(2-chlorophenylsulfonyl)-4-methylbenzamide (Gowda *et al.*, 2010*c*) display zero-dimensional architectures featuring inversion-related $R_2^2(8)$ dimers formed *via* N—H···O(S) hydrogen bonds, while strong N—H···O(S) hydrogen bonds in *N*-(2-chlorophenylsulfonyl)-4-nitrobenzamide (Suchetan *et al.*, 2011*d*) lead to one-dimensional chains. Similar to that observed in series 1, the methoxy- and fluoro-substituted compounds in series 2 show diversity in their intermolecular interactions. *N*-(2-chlorophenylsulfonyl)-4-methoxybenzamide (Sreenivasa *et al.*, 2014*b*) features structure-directing N—H···O(S) and C—H···O(S) hydrogen bonds and weak $\pi_{\rm aryl}$–$\pi_{\rm aryl}$ interactions, resulting in a two-dimensional structure. However, in *N*-(2-chlorophenylsulfonyl)-4-fluorobenzamide (II), N—H···O(S) and C—H···O(S) hydrogen bonds (with no structure-directing characteristics) between molecules form inversion-related dimers, and these dimers are interconnected *via* C═O···$\pi_{\rm aryl}$ interactions, forming a one-dimensional architecture.

Series 3: In series 3, the parent compound *N*-(4-chlorophenylsulfonyl)benzamide (Suchetan *et al.*, 2010*a*) crystallizes with two molecules in the asymmetric unit, while *N*-(4-chlorophenylsulfonyl)-4-chlorobenzamide (Suchetan *et al.*, 2010*b*), *N*-(4-chlorophenylsulfonyl)-4-methylbenzamide (Suchetan *et al.*, 2010*c*) and *N*-(4-chlorophenylsulfonyl)-4-nitrobenzamide (Suchetan *et al.*, 2011*a*) crystallize with one molecule, and *N*-(4-chlorophenylsulfonyl)-4-fluorobenzamide (III) crystallizes with one molecule and a molecule of water in the asymmetric unit. The values of the dihedral angle between the two aromatic rings in the molecules are in the range 62.8 (1)–89.5 (1)°, the smallest value being for *N*-(4-chlorophenylsulfonyl)benzamide and the largest for *N*-(4-chlorophenylsulfonyl)-4-methylbenzamide (Table 6). Except for compound (III), the crystals of all of the compounds feature N—H···O(S) hydrogen bonds, either forming $R_2^2(8)$ inversion dimers (zero-dimensional structure) or one-dimensional chains. Once again, the fluoro-substituted compound (III) displays a variety of hydrogen bonds and weak interactions (Tables 3 and 6), leading to a three-dimensional architecture.

5. Synthesis and crystallization

Compounds (I)–(III) were prepared by refluxing a mixture of 4-fluorobenzoic acid, the corresponding substituted benzenesulfonamides and phosphorousoxychloride 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 bicarbonate solutions. The compounds were later re-precipitated by acidifying the filtered solutions with dilute HCl. They were filtered,

Table 7
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{14}H_{12}FNO_3S$	$C_{13}H_9ClFNO_3S$	$C_{13}H_9ClFNO_3S\cdot H_2O$
M_r	293.31	313.72	331.74
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$
Temperature (K)	173	173	173
a, b, c (Å)	10.0259 (3), 12.4289 (3), 21.6241 (6)	7.9009 (2), 9.0775 (3), 18.4216 (5)	45.5989 (11), 4.8853 (1), 12.6517 (3)
β (°)	92.443 (1)	99.801 (1)	94.481 (1)
V (Å^3)	2692.15 (13)	1301.92 (6)	2809.73 (11)
Z	8	4	8
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	2.32	4.29	4.06
Crystal size (mm)	0.28 × 0.26 × 0.21	0.30 × 0.27 × 0.23	0.28 × 0.25 × 0.23
Data collection			
Diffractometer	Bruker APEXII	Bruker APEXII	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)	Multi-scan (*SADABS*; Bruker, 2009)	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.548, 0.614	0.317, 0.373	0.369, 0.393
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	26667, 4404, 4165	10032, 2124, 2074	11176, 2320, 2030
R_{int}	0.042	0.042	0.054
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.587	0.585	0.584
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.121, 1.00	0.041, 0.124, 0.97	0.039, 0.118, 0.94
No. of reflections	4404	2124	2320
No. of parameters	371	185	205
No. of restraints	0	0	2
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	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.46	0.37, −0.50	0.34, −0.35

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

dried and recrystallized. [Melting point (m.p.) of (I) = 410 K, (II) = 428 K and (III) = 456 K]. Prism-like, colourless single crystals of all three of the compounds were obtained from slow evaporation of the respective solutions of the compounds in methanol (with few drops of water).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. The H atoms of the NH groups in (I)–(III) were located in difference Fourier maps and freely refined. The H atoms of the water molecule in (III) were located in a difference Fourier map and were refined with the bond length restraint O—H = 0.83 (3) Å. The other H atoms were positioned with idealized geometry using a riding model: C—H = 0.93–0.96 Å, with $U_{iso} = 1.5U_{eq}$(C-methyl) and $1.2U_{eq}$(C) for other H atoms. In the final cycles of refinement, reflections (0 1 1), (0 0 2) and ($\bar{7}$ 0 20) in (I), (0 0 2) in (II) and (2 0 0) in (III) were omitted due to large differences in F^2_{obs} and F^2_{calc}.

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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*, *SAINT-Plus* and *XPREP*. 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. (2010*a*). *Acta Cryst.* E**66**, o747.

Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o794.

Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010*c*). *Acta Cryst.* E**66**, o1466.

Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.

Hamad, A. S. & Abed, F. S. (2014). *J. Appl. Chem*, **3**, 56–63.

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., De Dios, A., 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.

Manojkumar, K. E., Sreenivasa, S., Mohan, N. R., Madhu Chakrapani Rao, T. & Harikrishna, T. (2013). *J. Appl. Chem*, **2**, 730–737.

Mohan, N. R., Sreenivasa, S., Manojkumar, K. E. & Chakrapani Rao, T. M. (2013). *J. Appl. Chem.* **2**, 722–729.

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.* A**64**, 112–122.

Sreenivasa, S., Palakshamurthy, B. S., Madankumar, S., Lokanath, N. K. & Suchetan, P. A. (2014*a*). *Acta Cryst.* E**70**, o193.

Sreenivasa, S., Palakshamurthy, B. S., Pampa, K. J., Lokanath, N. K. & Suchetan, P. A. (2014*b*). *Acta Cryst.* E**70**, o199.

Suchetan, P. A., Foro, S. & Gowda, B. T. (2011*a*). *Acta Cryst.* E**67**, o904.

Suchetan, P. A., Foro, S. & Gowda, B. T. (2011*b*). *Acta Cryst.* E**67**, o929.

Suchetan, P. A., Foro, S. & Gowda, B. T. (2011*c*). *Acta Cryst.* E**67**, o146.

Suchetan, P. A., Foro, S. & Gowda, B. T. (2011*d*). *Acta Cryst.* E**67**, o930.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*a*). *Acta Cryst.* E**66**, o766.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o1253.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*c*). *Acta Cryst.* E**66**, o1501.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*d*). *Acta Cryst.* E**66**, o1024.

Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*e*). *Acta Cryst.* E**66**, o1997.

supporting information

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Crystal structures of three *N*-(arylsulfonyl)-4-fluorobenzamides

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

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (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) 4-Fluoro-*N*-(2-methylphenylsulfonyl)benzamide

Crystal data

$C_{14}H_{12}FNO_3S$
M_r = 293.31
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.0259 (3) Å
b = 12.4289 (3) Å
c = 21.6241 (6) Å
β = 92.443 (1)°
V = 2692.15 (13) Å^3
Z = 8
$F(000)$ = 1216
Prism
D_x = 1.447 Mg m^{-3}
Melting point: 406 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 147 reflections
θ = 5.4–64.8°
μ = 2.32 mm^{-1}
T = 173 K
Prism, colourless
0.28 × 0.26 × 0.21 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.548, T_{max} = 0.614
26667 measured reflections
4404 independent reflections
4165 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 64.8°, θ_{min} = 5.4°
$h = -11\rightarrow11$
$k = -14\rightarrow14$
$l = -25\rightarrow22$
1 standard reflections every 1 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.121
S = 1.00
4404 reflections
371 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

$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2 + 2.0561P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.36$ e Å^{-3}

$\Delta\rho_{min} = -0.46$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
H1B	0.025 (3)	0.916 (2)	0.1633 (11)	0.025 (6)*
H1A	0.141 (3)	1.144 (2)	0.0099 (15)	0.047 (9)*
S1B	0.19601 (5)	0.98360 (4)	0.12225 (2)	0.01914 (16)
S1A	0.23194 (5)	1.14938 (4)	−0.07809 (2)	0.02559 (17)
F1B	−0.25542 (13)	0.86212 (10)	0.41052 (6)	0.0328 (3)
O3B	0.15022 (14)	1.09200 (11)	0.24124 (6)	0.0235 (3)
F1A	−0.30717 (16)	1.30341 (12)	0.16831 (7)	0.0461 (4)
N1B	0.08517 (17)	0.95866 (14)	0.17484 (8)	0.0208 (4)
O2B	0.17274 (15)	0.90165 (12)	0.07688 (6)	0.0290 (4)
O1B	0.18579 (15)	1.09350 (12)	0.10231 (6)	0.0267 (3)
N1A	0.12947 (17)	1.17667 (15)	−0.02228 (8)	0.0232 (4)
C9B	−0.05607 (19)	0.86357 (15)	0.27587 (9)	0.0200 (4)
H9B	−0.0281	0.8166	0.2456	0.024*
C8A	−0.05155 (19)	1.26905 (17)	0.02721 (9)	0.0223 (4)
O3A	0.04337 (19)	1.32855 (18)	−0.06557 (9)	0.0580 (6)
C13B	−0.0598 (2)	1.04189 (16)	0.32012 (9)	0.0203 (4)
H13B	−0.0330	1.1135	0.3199	0.024*
C8B	−0.01651 (19)	0.97167 (15)	0.27488 (9)	0.0181 (4)
C11B	−0.1779 (2)	0.89869 (17)	0.36500 (9)	0.0223 (4)
C11A	−0.2225 (2)	1.29198 (18)	0.12140 (10)	0.0275 (5)
C2A	0.4432 (2)	1.27633 (19)	−0.03690 (10)	0.0289 (5)
C7B	0.07876 (19)	1.01470 (15)	0.23026 (9)	0.0187 (4)
C1B	0.35084 (19)	0.96772 (16)	0.16277 (8)	0.0191 (4)
O1A	0.15399 (18)	1.14550 (19)	−0.13481 (7)	0.0515 (6)
C10A	−0.1853 (2)	1.18949 (17)	0.10451 (10)	0.0257 (5)
H10A	−0.2181	1.1294	0.1245	0.031*
C12A	−0.1790 (2)	1.38297 (17)	0.09230 (11)	0.0284 (5)
H12A	−0.2062	1.4509	0.1046	0.034*
C2B	0.3912 (2)	0.86733 (17)	0.18633 (9)	0.0259 (5)
C10B	−0.1364 (2)	0.82636 (16)	0.32158 (9)	0.0218 (4)
H10B	−0.1618	0.7545	0.3230	0.026*

C1A	0.34173 (19)	1.25928 (16)	−0.08229 (9)	0.0215 (4)
C9A	−0.0986 (2)	1.17872 (16)	0.05733 (9)	0.0211 (4)
H9A	−0.0711	1.1106	0.0455	0.025*
C3B	0.5152 (2)	0.8644 (2)	0.21795 (11)	0.0357 (6)
H3B	0.5455	0.7996	0.2349	0.043*
C12B	−0.1426 (2)	1.00568 (16)	0.36547 (9)	0.0232 (4)
H12B	−0.1733	1.0523	0.3953	0.028*
C6B	0.4292 (2)	1.05960 (18)	0.16986 (9)	0.0246 (4)
H6B	0.3992	1.1251	0.1538	0.029*
C13A	−0.0943 (2)	1.37067 (17)	0.04461 (11)	0.0287 (5)
H13A	−0.0652	1.4310	0.0237	0.034*
C6A	0.3221 (2)	1.32776 (19)	−0.13289 (11)	0.0311 (5)
H6A	0.2538	1.3141	−0.1623	0.037*
C14B	0.3083 (3)	0.76662 (18)	0.17972 (12)	0.0382 (6)
H14A	0.3605	0.7058	0.1936	0.057*
H14B	0.2805	0.7568	0.1371	0.057*
H14C	0.2311	0.7730	0.2043	0.057*
C7A	0.0419 (2)	1.26305 (19)	−0.02419 (10)	0.0288 (5)
C5B	0.5520 (2)	1.0525 (2)	0.20096 (10)	0.0337 (5)
H5B	0.6059	1.1131	0.2057	0.040*
C4B	0.5943 (2)	0.9547 (2)	0.22491 (10)	0.0395 (6)
H4B	0.6769	0.9497	0.2459	0.047*
C5A	0.4038 (3)	1.4155 (2)	−0.13928 (14)	0.0440 (7)
H5A	0.3927	1.4605	−0.1735	0.053*
C4A	0.5015 (3)	1.4359 (2)	−0.09479 (16)	0.0519 (8)
H4A	0.5553	1.4964	−0.0983	0.062*
C3A	0.5214 (3)	1.3684 (2)	−0.04501 (14)	0.0457 (7)
H3A	0.5890	1.3843	−0.0156	0.055*
C14A	0.4705 (3)	1.2051 (3)	0.01773 (12)	0.0456 (7)
H14D	0.3884	1.1898	0.0372	0.068*
H14E	0.5308	1.2407	0.0467	0.068*
H14F	0.5099	1.1391	0.0044	0.068*
O2A	0.30147 (18)	1.05485 (12)	−0.05762 (8)	0.0405 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1B	0.0203 (3)	0.0232 (3)	0.0137 (3)	−0.00057 (17)	−0.00218 (19)	−0.00037 (17)
S1A	0.0249 (3)	0.0336 (3)	0.0185 (3)	−0.0111 (2)	0.0033 (2)	−0.0071 (2)
F1B	0.0362 (7)	0.0321 (7)	0.0311 (7)	−0.0018 (5)	0.0135 (6)	0.0065 (5)
O3B	0.0282 (8)	0.0217 (7)	0.0209 (7)	−0.0055 (6)	0.0024 (6)	−0.0018 (6)
F1A	0.0571 (9)	0.0440 (8)	0.0390 (8)	0.0092 (7)	0.0248 (7)	−0.0041 (6)
N1B	0.0195 (9)	0.0249 (9)	0.0178 (9)	−0.0062 (7)	−0.0031 (7)	−0.0036 (7)
O2B	0.0305 (8)	0.0377 (8)	0.0189 (7)	−0.0068 (6)	0.0001 (6)	−0.0088 (6)
O1B	0.0312 (8)	0.0285 (8)	0.0201 (7)	0.0048 (6)	−0.0012 (6)	0.0075 (6)
N1A	0.0223 (9)	0.0305 (9)	0.0165 (9)	−0.0020 (7)	−0.0024 (7)	0.0039 (7)
C9B	0.0208 (10)	0.0192 (9)	0.0197 (10)	0.0027 (7)	−0.0033 (8)	−0.0031 (8)
C8A	0.0173 (10)	0.0286 (11)	0.0205 (10)	0.0000 (8)	−0.0047 (8)	0.0042 (8)

O3A	0.0389 (10)	0.0834 (15)	0.0532 (12)	0.0299 (10)	0.0207 (9)	0.0507 (11)
C13B	0.0224 (10)	0.0176 (9)	0.0205 (10)	0.0005 (8)	−0.0023 (8)	−0.0007 (8)
C8B	0.0181 (10)	0.0196 (9)	0.0161 (9)	0.0007 (7)	−0.0037 (7)	0.0012 (7)
C11B	0.0197 (10)	0.0276 (11)	0.0198 (10)	−0.0001 (8)	0.0024 (8)	0.0059 (8)
C11A	0.0275 (11)	0.0335 (12)	0.0217 (11)	0.0016 (9)	0.0032 (9)	−0.0050 (9)
C2A	0.0183 (10)	0.0414 (13)	0.0270 (11)	−0.0027 (9)	0.0014 (8)	−0.0093 (10)
C7B	0.0200 (10)	0.0179 (9)	0.0176 (9)	0.0023 (8)	−0.0047 (8)	0.0003 (7)
C1B	0.0189 (10)	0.0258 (10)	0.0127 (9)	0.0017 (8)	0.0025 (7)	−0.0010 (7)
O1A	0.0365 (10)	0.0998 (16)	0.0184 (8)	−0.0369 (10)	0.0025 (7)	−0.0104 (9)
C10A	0.0288 (11)	0.0232 (10)	0.0254 (11)	−0.0040 (8)	0.0049 (9)	0.0004 (8)
C12A	0.0262 (11)	0.0227 (10)	0.0358 (12)	0.0030 (8)	−0.0039 (9)	−0.0040 (9)
C2B	0.0314 (12)	0.0284 (11)	0.0183 (10)	0.0089 (9)	0.0055 (9)	0.0000 (8)
C10B	0.0222 (10)	0.0184 (9)	0.0247 (11)	−0.0014 (8)	−0.0004 (8)	0.0033 (8)
C1A	0.0174 (10)	0.0240 (10)	0.0234 (10)	−0.0002 (8)	0.0040 (8)	−0.0025 (8)
C9A	0.0218 (10)	0.0218 (10)	0.0194 (10)	−0.0021 (8)	−0.0027 (8)	−0.0032 (8)
C3B	0.0346 (13)	0.0494 (14)	0.0234 (11)	0.0220 (11)	0.0030 (10)	0.0054 (10)
C12B	0.0261 (11)	0.0232 (10)	0.0202 (10)	0.0029 (8)	0.0021 (8)	−0.0028 (8)
C6B	0.0258 (11)	0.0305 (11)	0.0176 (10)	−0.0051 (9)	0.0027 (8)	−0.0011 (8)
C13A	0.0222 (11)	0.0251 (11)	0.0382 (13)	−0.0008 (8)	−0.0042 (9)	0.0096 (9)
C6A	0.0252 (11)	0.0387 (12)	0.0300 (12)	0.0094 (9)	0.0099 (9)	0.0066 (10)
C14B	0.0574 (16)	0.0207 (11)	0.0365 (13)	0.0065 (10)	0.0022 (11)	0.0030 (9)
C7A	0.0192 (10)	0.0428 (13)	0.0242 (11)	0.0026 (9)	−0.0008 (8)	0.0121 (10)
C5B	0.0235 (11)	0.0565 (15)	0.0214 (11)	−0.0125 (10)	0.0048 (9)	−0.0069 (10)
C4B	0.0188 (11)	0.0783 (19)	0.0213 (11)	0.0079 (12)	−0.0006 (9)	−0.0022 (12)
C5A	0.0447 (15)	0.0306 (13)	0.0590 (17)	0.0096 (11)	0.0297 (13)	0.0109 (12)
C4A	0.0470 (16)	0.0310 (13)	0.081 (2)	−0.0122 (12)	0.0373 (16)	−0.0114 (14)
C3A	0.0278 (13)	0.0561 (16)	0.0536 (17)	−0.0163 (11)	0.0080 (11)	−0.0263 (14)
C14A	0.0313 (13)	0.0746 (19)	0.0301 (13)	0.0010 (12)	−0.0100 (10)	0.0012 (13)
O2A	0.0515 (11)	0.0221 (8)	0.0496 (10)	−0.0010 (7)	0.0231 (8)	−0.0059 (7)

Geometric parameters (Å, º)

S1B—O2B	1.4265 (15)	C1B—C6B	1.391 (3)
S1B—O1B	1.4347 (15)	C1B—C2B	1.401 (3)
S1B—N1B	1.6528 (18)	C10A—C9A	1.375 (3)
S1B—C1B	1.7609 (19)	C10A—H10A	0.9300
S1A—O2A	1.4268 (18)	C12A—C13A	1.372 (3)
S1A—O1A	1.4268 (17)	C12A—H12A	0.9300
S1A—N1A	1.6531 (18)	C2B—C3B	1.393 (3)
S1A—C1A	1.759 (2)	C2B—C14B	1.506 (3)
F1B—C11B	1.358 (2)	C10B—H10B	0.9300
O3B—C7B	1.216 (2)	C1A—C6A	1.394 (3)
F1A—C11A	1.358 (3)	C9A—H9A	0.9300
N1B—C7B	1.390 (3)	C3B—C4B	1.379 (4)
N1B—H1B	0.83 (3)	C3B—H3B	0.9300
N1A—C7A	1.386 (3)	C12B—H12B	0.9300
N1A—H1A	0.81 (3)	C6B—C5B	1.381 (3)
C9B—C10B	1.381 (3)	C6B—H6B	0.9300

C9B—C8B	1.401 (3)	C13A—H13A	0.9300
C9B—H9B	0.9300	C6A—C5A	1.374 (4)
C8A—C9A	1.390 (3)	C6A—H6A	0.9300
C8A—C13A	1.391 (3)	C14B—H14A	0.9600
C8A—C7A	1.486 (3)	C14B—H14B	0.9600
O3A—C7A	1.210 (3)	C14B—H14C	0.9600
C13B—C12B	1.387 (3)	C5B—C4B	1.381 (4)
C13B—C8B	1.394 (3)	C5B—H5B	0.9300
C13B—H13B	0.9300	C4B—H4B	0.9300
C8B—C7B	1.486 (3)	C5A—C4A	1.368 (4)
C11B—C12B	1.376 (3)	C5A—H5A	0.9300
C11B—C10B	1.377 (3)	C4A—C3A	1.373 (4)
C11A—C12A	1.374 (3)	C4A—H4A	0.9300
C11A—C10A	1.381 (3)	C3A—H3A	0.9300
C2A—C1A	1.400 (3)	C14A—H14D	0.9600
C2A—C3A	1.402 (4)	C14A—H14E	0.9600
C2A—C14A	1.492 (4)	C14A—H14F	0.9600

O2B—S1B—O1B	117.75 (9)	C11B—C10B—C9B	118.15 (18)
O2B—S1B—N1B	104.14 (9)	C11B—C10B—H10B	120.9
O1B—S1B—N1B	110.11 (9)	C9B—C10B—H10B	120.9
O2B—S1B—C1B	112.15 (9)	C6A—C1A—C2A	122.2 (2)
O1B—S1B—C1B	107.84 (9)	C6A—C1A—S1A	116.56 (16)
N1B—S1B—C1B	103.94 (9)	C2A—C1A—S1A	121.28 (17)
O2A—S1A—O1A	118.77 (12)	C10A—C9A—C8A	120.44 (19)
O2A—S1A—N1A	104.66 (10)	C10A—C9A—H9A	119.8
O1A—S1A—N1A	107.45 (10)	C8A—C9A—H9A	119.8
O2A—S1A—C1A	110.91 (10)	C4B—C3B—C2B	121.9 (2)
O1A—S1A—C1A	107.63 (11)	C4B—C3B—H3B	119.0
N1A—S1A—C1A	106.75 (9)	C2B—C3B—H3B	119.0
C7B—N1B—S1B	123.81 (15)	C11B—C12B—C13B	118.04 (19)
C7B—N1B—H1B	121.1 (17)	C11B—C12B—H12B	121.0
S1B—N1B—H1B	114.4 (17)	C13B—C12B—H12B	121.0
C7A—N1A—S1A	123.34 (15)	C5B—C6B—C1B	119.3 (2)
C7A—N1A—H1A	119 (2)	C5B—C6B—H6B	120.4
S1A—N1A—H1A	117 (2)	C1B—C6B—H6B	120.4
C10B—C9B—C8B	120.41 (18)	C12A—C13A—C8A	120.8 (2)
C10B—C9B—H9B	119.8	C12A—C13A—H13A	119.6
C8B—C9B—H9B	119.8	C8A—C13A—H13A	119.6
C9A—C8A—C13A	119.5 (2)	C5A—C6A—C1A	119.9 (2)
C9A—C8A—C7A	123.17 (19)	C5A—C6A—H6A	120.0
C13A—C8A—C7A	117.36 (19)	C1A—C6A—H6A	120.0
C12B—C13B—C8B	120.45 (18)	C2B—C14B—H14A	109.5
C12B—C13B—H13B	119.8	C2B—C14B—H14B	109.5
C8B—C13B—H13B	119.8	H14A—C14B—H14B	109.5
C13B—C8B—C9B	119.51 (19)	C2B—C14B—H14C	109.5
C13B—C8B—C7B	117.28 (17)	H14A—C14B—H14C	109.5
C9B—C8B—C7B	123.05 (18)	H14B—C14B—H14C	109.5

F1B—C11B—C12B	118.24 (18)	O3A—C7A—N1A	121.0 (2)
F1B—C11B—C10B	118.33 (18)	O3A—C7A—C8A	123.2 (2)
C12B—C11B—C10B	123.41 (19)	N1A—C7A—C8A	115.80 (18)
F1A—C11A—C12A	118.48 (19)	C4B—C5B—C6B	119.5 (2)
F1A—C11A—C10A	118.6 (2)	C4B—C5B—H5B	120.3
C12A—C11A—C10A	122.9 (2)	C6B—C5B—H5B	120.3
C1A—C2A—C3A	115.4 (2)	C5B—C4B—C3B	120.7 (2)
C1A—C2A—C14A	124.7 (2)	C5B—C4B—H4B	119.7
C3A—C2A—C14A	119.9 (2)	C3B—C4B—H4B	119.7
O3B—C7B—N1B	121.05 (19)	C4A—C5A—C6A	119.3 (2)
O3B—C7B—C8B	123.19 (17)	C4A—C5A—H5A	120.4
N1B—C7B—C8B	115.73 (16)	C6A—C5A—H5A	120.4
C6B—C1B—C2B	122.55 (19)	C5A—C4A—C3A	120.9 (2)
C6B—C1B—S1B	116.40 (15)	C5A—C4A—H4A	119.6
C2B—C1B—S1B	121.05 (16)	C3A—C4A—H4A	119.6
C9A—C10A—C11A	118.20 (19)	C4A—C3A—C2A	122.3 (2)
C9A—C10A—H10A	120.9	C4A—C3A—H3A	118.9
C11A—C10A—H10A	120.9	C2A—C3A—H3A	118.9
C13A—C12A—C11A	118.1 (2)	C2A—C14A—H14D	109.5
C13A—C12A—H12A	120.9	C2A—C14A—H14E	109.5
C11A—C12A—H12A	120.9	H14D—C14A—H14E	109.5
C3B—C2B—C1B	116.1 (2)	C2A—C14A—H14F	109.5
C3B—C2B—C14B	120.1 (2)	H14D—C14A—H14F	109.5
C1B—C2B—C14B	123.77 (19)	H14E—C14A—H14F	109.5

O2B—S1B—N1B—C7B	174.96 (16)	C14A—C2A—C1A—S1A	1.0 (3)
O1B—S1B—N1B—C7B	−57.91 (18)	O2A—S1A—C1A—C6A	139.74 (16)
C1B—S1B—N1B—C7B	57.37 (18)	O1A—S1A—C1A—C6A	8.3 (2)
O2A—S1A—N1A—C7A	−179.46 (17)	N1A—S1A—C1A—C6A	−106.81 (17)
O1A—S1A—N1A—C7A	−52.3 (2)	O2A—S1A—C1A—C2A	−40.3 (2)
C1A—S1A—N1A—C7A	62.90 (19)	O1A—S1A—C1A—C2A	−171.72 (18)
C12B—C13B—C8B—C9B	−0.7 (3)	N1A—S1A—C1A—C2A	73.19 (19)
C12B—C13B—C8B—C7B	−176.25 (17)	C11A—C10A—C9A—C8A	−0.8 (3)
C10B—C9B—C8B—C13B	−0.6 (3)	C13A—C8A—C9A—C10A	−0.8 (3)
C10B—C9B—C8B—C7B	174.72 (17)	C7A—C8A—C9A—C10A	−179.80 (18)
S1B—N1B—C7B—O3B	5.4 (3)	C1B—C2B—C3B—C4B	−0.8 (3)
S1B—N1B—C7B—C8B	−172.64 (13)	C14B—C2B—C3B—C4B	179.9 (2)
C13B—C8B—C7B—O3B	23.1 (3)	F1B—C11B—C12B—C13B	177.87 (17)
C9B—C8B—C7B—O3B	−152.28 (19)	C10B—C11B—C12B—C13B	−0.6 (3)
C13B—C8B—C7B—N1B	−158.91 (17)	C8B—C13B—C12B—C11B	1.3 (3)
C9B—C8B—C7B—N1B	25.7 (3)	C2B—C1B—C6B—C5B	0.3 (3)
O2B—S1B—C1B—C6B	133.35 (16)	S1B—C1B—C6B—C5B	−179.72 (16)
O1B—S1B—C1B—C6B	2.11 (18)	C11A—C12A—C13A—C8A	−1.4 (3)
N1B—S1B—C1B—C6B	−114.76 (16)	C9A—C8A—C13A—C12A	2.0 (3)
O2B—S1B—C1B—C2B	−46.72 (19)	C7A—C8A—C13A—C12A	−178.98 (18)
O1B—S1B—C1B—C2B	−177.95 (16)	C2A—C1A—C6A—C5A	−0.1 (3)
N1B—S1B—C1B—C2B	65.17 (18)	S1A—C1A—C6A—C5A	179.88 (17)
F1A—C11A—C10A—C9A	−179.39 (18)	S1A—N1A—C7A—O3A	−5.6 (3)

C12A—C11A—C10A—C9A	1.5 (3)	S1A—N1A—C7A—C8A	174.40 (14)
F1A—C11A—C12A—C13A	−179.49 (19)	C9A—C8A—C7A—O3A	149.7 (2)
C10A—C11A—C12A—C13A	−0.3 (3)	C13A—C8A—C7A—O3A	−29.2 (3)
C6B—C1B—C2B—C3B	0.4 (3)	C9A—C8A—C7A—N1A	−30.3 (3)
S1B—C1B—C2B—C3B	−179.57 (16)	C13A—C8A—C7A—N1A	150.71 (19)
C6B—C1B—C2B—C14B	179.6 (2)	C1B—C6B—C5B—C4B	−0.6 (3)
S1B—C1B—C2B—C14B	−0.3 (3)	C6B—C5B—C4B—C3B	0.1 (3)
F1B—C11B—C10B—C9B	−179.09 (17)	C2B—C3B—C4B—C5B	0.6 (4)
C12B—C11B—C10B—C9B	−0.6 (3)	C1A—C6A—C5A—C4A	−1.7 (3)
C8B—C9B—C10B—C11B	1.2 (3)	C6A—C5A—C4A—C3A	1.9 (4)
C3A—C2A—C1A—C6A	1.6 (3)	C5A—C4A—C3A—C2A	−0.3 (4)
C14A—C2A—C1A—C6A	−179.0 (2)	C1A—C2A—C3A—C4A	−1.4 (4)
C3A—C2A—C1A—S1A	−178.41 (17)	C14A—C2A—C3A—C4A	179.2 (2)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the fluorobenzene ring of molecule *B* of (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O1*B*	0.81 (3)	2.12 (3)	2.918 (2)	167 (2)
N1*B*—H1*B*···O1*A*^{i}	0.83 (3)	2.02 (3)	2.828 (3)	162 (3)
C6*A*—H6*A*···O3*B*	0.93	2.57	3.313 (3)	137
C10*B*—H10*B*···O1*B*^{ii}	0.93	2.59	3.376 (2)	143
C10*B*—H10*B*···O3*B*^{ii}	0.93	2.46	3.215 (2)	139
C4*B*—H4*B*···*Cg*^{iii}	0.93	2.72	3.646 (2)	173
C14*B*—H14*B*···O2*B*	0.96	2.45	3.058 (3)	121

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

(II) *N*-(2-Chlorophenylsulfonyl)-4-fluorobenzamide

Crystal data

$C_{13}H_9ClFNO_3S$	Prism
M_r = 313.72	D_x = 1.601 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 428 K
Hall symbol: -P 2ybc	Cu *Kα* radiation, λ = 1.54178 Å
a = 7.9009 (2) Å	Cell parameters from 133 reflections
b = 9.0775 (3) Å	θ = 5.5–64.3°
c = 18.4216 (5) Å	μ = 4.29 mm^{-1}
β = 99.801 (1)°	T = 173 K
V = 1301.92 (6) Å^3	Prism, colourless
Z = 4	0.30 × 0.27 × 0.23 mm
F(000) = 640	

Data collection

Bruker APEXII diffractometer	T_{min} = 0.317, T_{max} = 0.373
Radiation source: fine-focus sealed tube	10032 measured reflections
Graphite monochromator	2124 independent reflections
phi and φ scans	2074 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	R_{int} = 0.042
	θ_{max} = 64.3°, θ_{min} = 5.5°
	h = −9→9

$k = -10 \rightarrow 10$
$l = -21 \rightarrow 21$

1 standard reflections every 1 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.124$
$S = 0.97$
2124 reflections
185 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
$w = 1/[\sigma^2(F_o^2) + (0.0987P)^2 + 0.7647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
H1	0.410 (3)	0.844 (3)	0.5009 (14)	0.024 (6)*
S1	0.37229 (6)	0.89119 (5)	0.38590 (2)	0.0164 (2)
Cl1	0.16839 (7)	1.11686 (6)	0.47955 (3)	0.0297 (2)
F1	0.34792 (18)	0.38611 (13)	0.74018 (7)	0.0273 (3)
O3	0.2088 (2)	0.60943 (15)	0.41128 (8)	0.0271 (4)
O1	0.41851 (18)	0.79617 (16)	0.33118 (7)	0.0233 (4)
O2	0.47707 (16)	1.01786 (16)	0.40831 (7)	0.0209 (3)
N1	0.3743 (2)	0.79899 (19)	0.46309 (9)	0.0189 (4)
C8	0.3185 (3)	0.5908 (2)	0.54010 (11)	0.0177 (4)
C6	0.0748 (3)	0.8957 (2)	0.28779 (12)	0.0213 (5)
H6	0.1296	0.8275	0.2621	0.026*
C7	0.2942 (2)	0.6622 (2)	0.46614 (10)	0.0190 (4)
C5	−0.0885 (3)	0.9447 (2)	0.25822 (12)	0.0263 (5)
H5	−0.1431	0.9100	0.2127	0.032*
C1	0.1572 (2)	0.9480 (2)	0.35573 (10)	0.0168 (4)
C2	0.0741 (3)	1.0495 (2)	0.39430 (11)	0.0208 (5)
C12	0.4439 (3)	0.5719 (2)	0.66854 (11)	0.0215 (4)
H12	0.5198	0.6053	0.7094	0.026*
C4	−0.1698 (3)	1.0460 (2)	0.29697 (13)	0.0284 (5)
H4	−0.2791	1.0796	0.2771	0.034*
C13	0.4350 (3)	0.6399 (2)	0.60076 (11)	0.0199 (4)
H13	0.5071	0.7188	0.5956	0.024*

C9	0.2136 (3)	0.4697 (2)	0.54802 (12)	0.0238 (5)
H9	0.1372	0.4353	0.5075	0.029*
C11	0.3375 (3)	0.4534 (2)	0.67409 (11)	0.0210 (5)
C10	0.2222 (3)	0.4010 (2)	0.61510 (13)	0.0258 (5)
H10	0.1517	0.3211	0.6205	0.031*
C3	−0.0903 (3)	1.0973 (2)	0.36473 (13)	0.0276 (5)
H3	−0.1467	1.1639	0.3906	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0164 (3)	0.0194 (3)	0.0127 (3)	0.00182 (16)	0.0000 (2)	0.00090 (16)
Cl1	0.0282 (4)	0.0347 (4)	0.0244 (4)	0.0040 (2)	−0.0002 (2)	−0.01415 (19)
F1	0.0376 (8)	0.0273 (7)	0.0179 (7)	0.0058 (5)	0.0079 (5)	0.0058 (4)
O3	0.0339 (9)	0.0251 (8)	0.0192 (8)	−0.0037 (6)	−0.0047 (6)	−0.0008 (6)
O1	0.0243 (7)	0.0277 (8)	0.0178 (7)	0.0068 (6)	0.0031 (6)	−0.0004 (6)
O2	0.0187 (7)	0.0255 (8)	0.0172 (7)	−0.0037 (6)	−0.0010 (5)	0.0029 (5)
N1	0.0227 (9)	0.0206 (9)	0.0115 (8)	0.0006 (7)	−0.0026 (6)	−0.0001 (7)
C8	0.0190 (10)	0.0159 (9)	0.0174 (10)	0.0045 (7)	0.0006 (8)	−0.0007 (7)
C6	0.0235 (11)	0.0234 (11)	0.0163 (10)	−0.0021 (8)	0.0012 (8)	0.0001 (7)
C7	0.0200 (10)	0.0185 (10)	0.0178 (10)	0.0040 (8)	0.0010 (8)	−0.0008 (8)
C5	0.0243 (11)	0.0307 (12)	0.0207 (10)	−0.0045 (9)	−0.0049 (8)	0.0016 (9)
C1	0.0167 (9)	0.0175 (10)	0.0149 (9)	−0.0001 (7)	−0.0009 (7)	0.0030 (7)
C2	0.0215 (10)	0.0197 (11)	0.0197 (10)	0.0001 (8)	−0.0008 (8)	−0.0011 (8)
C12	0.0229 (10)	0.0228 (10)	0.0171 (10)	0.0041 (8)	−0.0011 (8)	−0.0013 (8)
C4	0.0185 (10)	0.0284 (12)	0.0350 (12)	0.0025 (9)	−0.0049 (9)	0.0064 (9)
C13	0.0195 (10)	0.0175 (10)	0.0223 (10)	0.0015 (8)	0.0028 (8)	0.0007 (8)
C9	0.0264 (11)	0.0194 (10)	0.0231 (10)	−0.0004 (8)	−0.0028 (8)	−0.0009 (8)
C11	0.0261 (10)	0.0208 (10)	0.0175 (10)	0.0095 (8)	0.0076 (8)	0.0033 (8)
C10	0.0280 (12)	0.0205 (11)	0.0290 (12)	−0.0021 (8)	0.0050 (9)	0.0038 (8)
C3	0.0233 (11)	0.0238 (12)	0.0348 (13)	0.0051 (8)	0.0029 (10)	−0.0016 (9)

Geometric parameters (Å, º)

S1—O1	1.4208 (14)	C5—C4	1.387 (3)
S1—O2	1.4358 (15)	C5—H5	0.9300
S1—N1	1.6478 (16)	C1—C2	1.395 (3)
S1—C1	1.7721 (19)	C2—C3	1.390 (3)
Cl1—C2	1.731 (2)	C12—C11	1.380 (3)
F1—C11	1.352 (2)	C12—C13	1.384 (3)
O3—C7	1.214 (3)	C12—H12	0.9300
N1—C7	1.399 (3)	C4—C3	1.379 (3)
N1—H1	0.82 (3)	C4—H4	0.9300
C8—C13	1.394 (3)	C13—H13	0.9300
C8—C9	1.399 (3)	C9—C10	1.375 (3)
C8—C7	1.492 (3)	C9—H9	0.9300
C6—C5	1.385 (3)	C11—C10	1.378 (3)
C6—C1	1.392 (3)	C10—H10	0.9300

C6—H6	0.9300	C3—H3	0.9300

O1—S1—O2	119.01 (9)	C3—C2—C1	119.76 (19)
O1—S1—N1	109.76 (9)	C3—C2—Cl1	118.34 (16)
O2—S1—N1	103.79 (8)	C1—C2—Cl1	121.90 (15)
O1—S1—C1	107.61 (9)	C11—C12—C13	118.46 (18)
O2—S1—C1	109.42 (9)	C11—C12—H12	120.8
N1—S1—C1	106.62 (9)	C13—C12—H12	120.8
C7—N1—S1	123.20 (14)	C3—C4—C5	120.64 (19)
C7—N1—H1	120.2 (18)	C3—C4—H4	119.7
S1—N1—H1	115.7 (18)	C5—C4—H4	119.7
C13—C8—C9	119.30 (19)	C12—C13—C8	120.40 (19)
C13—C8—C7	123.92 (18)	C12—C13—H13	119.8
C9—C8—C7	116.77 (18)	C8—C13—H13	119.8
C5—C6—C1	120.4 (2)	C10—C9—C8	120.7 (2)
C5—C6—H6	119.8	C10—C9—H9	119.7
C1—C6—H6	119.8	C8—C9—H9	119.7
O3—C7—N1	120.53 (18)	F1—C11—C10	119.02 (19)
O3—C7—C8	123.75 (18)	F1—C11—C12	118.31 (18)
N1—C7—C8	115.71 (16)	C10—C11—C12	122.67 (19)
C6—C5—C4	119.5 (2)	C9—C10—C11	118.5 (2)
C6—C5—H5	120.2	C9—C10—H10	120.8
C4—C5—H5	120.2	C11—C10—H10	120.8
C6—C1—C2	119.64 (18)	C4—C3—C2	120.1 (2)
C6—C1—S1	117.37 (15)	C4—C3—H3	120.0
C2—C1—S1	122.82 (15)	C2—C3—H3	120.0

O1—S1—N1—C7	−48.40 (17)	C6—C1—C2—C3	0.2 (3)
O2—S1—N1—C7	−176.62 (15)	S1—C1—C2—C3	175.27 (16)
C1—S1—N1—C7	67.89 (17)	C6—C1—C2—Cl1	179.12 (15)
S1—N1—C7—O3	−4.9 (3)	S1—C1—C2—Cl1	−5.8 (3)
S1—N1—C7—C8	176.37 (13)	C6—C5—C4—C3	−0.5 (3)
C13—C8—C7—O3	170.6 (2)	C11—C12—C13—C8	1.2 (3)
C9—C8—C7—O3	−11.0 (3)	C9—C8—C13—C12	−1.5 (3)
C13—C8—C7—N1	−10.7 (3)	C7—C8—C13—C12	176.78 (18)
C9—C8—C7—N1	167.68 (17)	C13—C8—C9—C10	1.2 (3)
C1—C6—C5—C4	−0.3 (3)	C7—C8—C9—C10	−177.21 (19)
C5—C6—C1—C2	0.4 (3)	C13—C12—C11—F1	179.23 (17)
C5—C6—C1—S1	−174.92 (16)	C13—C12—C11—C10	−0.5 (3)
O1—S1—C1—C6	−0.75 (18)	C8—C9—C10—C11	−0.6 (3)
O2—S1—C1—C6	129.90 (15)	F1—C11—C10—C9	−179.53 (18)
N1—S1—C1—C6	−118.45 (16)	C12—C11—C10—C9	0.2 (3)
O1—S1—C1—C2	−175.94 (16)	C5—C4—C3—C2	1.1 (3)
O2—S1—C1—C2	−45.29 (19)	C1—C2—C3—C4	−0.9 (3)
N1—S1—C1—C2	66.36 (18)	Cl1—C2—C3—C4	−179.92 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.82 (3)	2.16 (3)	2.968 (2)	172 (2)
C13—H13···O2^{i}	0.93	2.40	3.194 (2)	144

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

(III) *N*-(4-Chlorophenylsulfonyl)-4-fluorobenzamide monohydrate

Crystal data

$C_{13}H_9ClFNO_3S\cdot H_2O$
M_r = 331.74
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 45.5989 (11) Å
b = 4.8853 (1) Å
c = 12.6517 (3) Å
β = 94.481 (1)°
V = 2809.73 (11) Å^3
Z = 8
F(000) = 1360
Prism
D_x = 1.568 Mg m^{-3}
Melting point: 456 K
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 163 reflections
θ = 5.8–64.3°
μ = 4.06 mm^{-1}
T = 173 K
Prism, colourless
0.28 × 0.25 × 0.23 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and *φ* scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.369, T_{max} = 0.393
11176 measured reflections
2320 independent reflections
2030 reflections with *I* > 2*σ*(*I*)
R_{int} = 0.054
θ_{max} = 64.3°, θ_{min} = 5.8°
h = −52→51
k = −5→5
l = −14→14
1 standard reflections every 1 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.118
S = 0.94
2320 reflections
205 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
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2 + 2.087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 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)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.356285 (11)	0.64551 (10)	0.55127 (4)	0.0147 (2)
Cl1	0.261887 (12)	−0.14924 (12)	0.34233 (4)	0.0273 (2)
O2	0.36322 (3)	0.8548 (3)	0.47847 (11)	0.0176 (4)
O1	0.34790 (3)	0.7192 (3)	0.65445 (11)	0.0195 (4)
F1	0.49577 (3)	−0.3420 (3)	0.62191 (12)	0.0384 (4)
O3	0.39675 (3)	0.4091 (3)	0.40288 (11)	0.0207 (4)
O4	0.38058 (4)	0.1618 (4)	0.75835 (13)	0.0290 (4)
N1	0.38535 (4)	0.4484 (4)	0.57495 (14)	0.0161 (4)
C7	0.40210 (5)	0.3508 (4)	0.49595 (16)	0.0165 (5)
C8	0.42713 (5)	0.1710 (4)	0.53341 (17)	0.0174 (5)
C3	0.28843 (5)	0.1252 (5)	0.50950 (18)	0.0216 (5)
H3	0.2753	0.0371	0.5507	0.026*
C13	0.43853 (5)	−0.0018 (5)	0.45932 (17)	0.0218 (5)
H13	0.4305	−0.0004	0.3895	0.026*
C2	0.30931 (5)	0.3027 (5)	0.55508 (17)	0.0206 (5)
H2	0.3105	0.3346	0.6278	0.025*
C1	0.32848 (4)	0.4331 (4)	0.49134 (16)	0.0155 (5)
C6	0.32692 (5)	0.3910 (5)	0.38239 (16)	0.0174 (5)
H6	0.3397	0.4821	0.3406	0.021*
C5	0.30619 (5)	0.2126 (5)	0.33712 (16)	0.0189 (5)
H5	0.3049	0.1814	0.2644	0.023*
C4	0.28742 (5)	0.0806 (5)	0.40070 (17)	0.0190 (5)
C9	0.43954 (5)	0.1702 (5)	0.63795 (18)	0.0241 (5)
H9	0.4321	0.2858	0.6878	0.029*
C10	0.46276 (5)	−0.0009 (6)	0.66765 (19)	0.0295 (6)
H10	0.4712	−0.0013	0.7369	0.035*
C12	0.46171 (5)	−0.1755 (5)	0.48843 (19)	0.0265 (6)
H12	0.4694	−0.2917	0.4393	0.032*
C11	0.47306 (5)	−0.1710 (5)	0.5923 (2)	0.0264 (6)
H1O4	0.3743 (8)	0.011 (6)	0.761 (3)	0.067 (12)*
H1	0.3854 (6)	0.373 (6)	0.634 (2)	0.037 (8)*
H2O4	0.3812 (7)	0.229 (6)	0.8178 (19)	0.039 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0164 (3)	0.0130 (3)	0.0148 (3)	0.0011 (2)	0.0022 (2)	−0.00059 (18)
Cl1	0.0245 (4)	0.0239 (4)	0.0326 (4)	−0.0091 (2)	−0.0040 (3)	0.0024 (2)
O2	0.0191 (8)	0.0126 (8)	0.0213 (8)	0.0000 (6)	0.0024 (6)	0.0004 (6)
O1	0.0221 (8)	0.0194 (8)	0.0173 (7)	0.0012 (7)	0.0040 (6)	−0.0034 (6)

F1	0.0277 (8)	0.0398 (10)	0.0466 (9)	0.0179 (7)	−0.0037 (7)	0.0018 (7)
O3	0.0240 (8)	0.0209 (8)	0.0173 (8)	0.0031 (7)	0.0034 (6)	0.0016 (6)
O4	0.0504 (12)	0.0206 (11)	0.0165 (8)	−0.0034 (8)	0.0067 (8)	−0.0021 (7)
N1	0.0180 (10)	0.0154 (10)	0.0149 (9)	0.0003 (8)	0.0004 (7)	0.0021 (7)
C7	0.0165 (11)	0.0135 (11)	0.0195 (11)	−0.0041 (8)	0.0010 (8)	−0.0018 (8)
C8	0.0145 (11)	0.0155 (11)	0.0225 (11)	−0.0025 (8)	0.0030 (8)	0.0011 (8)
C3	0.0181 (11)	0.0233 (13)	0.0240 (12)	−0.0016 (9)	0.0048 (9)	0.0060 (9)
C13	0.0210 (11)	0.0217 (13)	0.0227 (11)	0.0006 (10)	0.0017 (9)	−0.0018 (9)
C2	0.0211 (12)	0.0232 (13)	0.0175 (11)	0.0012 (10)	0.0022 (9)	0.0013 (9)
C1	0.0156 (11)	0.0114 (11)	0.0195 (10)	0.0029 (9)	0.0014 (8)	0.0003 (8)
C6	0.0159 (11)	0.0176 (12)	0.0191 (10)	0.0016 (9)	0.0031 (8)	0.0031 (9)
C5	0.0183 (11)	0.0213 (12)	0.0168 (10)	0.0010 (9)	0.0001 (8)	−0.0011 (9)
C4	0.0158 (11)	0.0133 (11)	0.0273 (11)	−0.0003 (9)	−0.0024 (8)	0.0010 (9)
C9	0.0224 (12)	0.0268 (14)	0.0230 (11)	0.0033 (10)	0.0005 (9)	−0.0032 (9)
C10	0.0225 (12)	0.0397 (16)	0.0255 (12)	0.0043 (11)	−0.0040 (9)	0.0016 (11)
C12	0.0227 (12)	0.0236 (14)	0.0335 (13)	0.0022 (10)	0.0045 (10)	−0.0069 (10)
C11	0.0163 (12)	0.0251 (14)	0.0374 (13)	0.0061 (10)	−0.0016 (10)	0.0050 (10)

Geometric parameters (Å, º)

S1—O2	1.4279 (15)	C3—H3	0.9300
S1—O1	1.4346 (14)	C13—C12	1.382 (3)
S1—N1	1.6466 (18)	C13—H13	0.9300
S1—C1	1.763 (2)	C2—C1	1.389 (3)
Cl1—C4	1.740 (2)	C2—H2	0.9300
F1—C11	1.360 (3)	C1—C6	1.390 (3)
O3—C7	1.217 (3)	C6—C5	1.377 (3)
O4—H1O4	0.79 (3)	C6—H6	0.9300
O4—H2O4	0.82 (2)	C5—C4	1.380 (3)
N1—C7	1.389 (3)	C5—H5	0.9300
N1—H1	0.84 (3)	C9—C10	1.378 (3)
C7—C8	1.488 (3)	C9—H9	0.9300
C8—C13	1.392 (3)	C10—C11	1.375 (4)
C8—C9	1.397 (3)	C10—H10	0.9300
C3—C2	1.380 (3)	C12—C11	1.374 (3)
C3—C4	1.391 (3)	C12—H12	0.9300
O2—S1—O1	119.70 (9)	C2—C1—C6	121.5 (2)
O2—S1—N1	108.70 (9)	C2—C1—S1	118.95 (16)
O1—S1—N1	104.43 (9)	C6—C1—S1	119.53 (16)
O2—S1—C1	109.39 (9)	C5—C6—C1	119.1 (2)
O1—S1—C1	107.77 (9)	C5—C6—H6	120.4
N1—S1—C1	105.99 (10)	C1—C6—H6	120.4
H1O4—O4—H2O4	109 (3)	C6—C5—C4	119.3 (2)
C7—N1—S1	123.36 (15)	C6—C5—H5	120.3
C7—N1—H1	122 (2)	C4—C5—H5	120.3
S1—N1—H1	112 (2)	C5—C4—C3	122.0 (2)
O3—C7—N1	122.4 (2)	C5—C4—Cl1	118.63 (17)

O3—C7—C8	122.48 (19)	C3—C4—Cl1	119.37 (17)
N1—C7—C8	115.13 (18)	C10—C9—C8	120.4 (2)
C13—C8—C9	119.4 (2)	C10—C9—H9	119.8
C13—C8—C7	117.45 (19)	C8—C9—H9	119.8
C9—C8—C7	123.2 (2)	C9—C10—C11	118.3 (2)
C2—C3—C4	118.7 (2)	C9—C10—H10	120.8
C2—C3—H3	120.6	C11—C10—H10	120.8
C4—C3—H3	120.6	C11—C12—C13	117.9 (2)
C12—C13—C8	120.7 (2)	C11—C12—H12	121.0
C12—C13—H13	119.6	C13—C12—H12	121.0
C8—C13—H13	119.6	F1—C11—C12	118.4 (2)
C3—C2—C1	119.4 (2)	F1—C11—C10	118.3 (2)
C3—C2—H2	120.3	C12—C11—C10	123.3 (2)
C1—C2—H2	120.3		

O2—S1—N1—C7	−45.29 (19)	O1—S1—C1—C6	163.03 (17)
O1—S1—N1—C7	−174.12 (17)	N1—S1—C1—C6	−85.63 (19)
C1—S1—N1—C7	72.20 (19)	C2—C1—C6—C5	−1.0 (3)
S1—N1—C7—O3	1.5 (3)	S1—C1—C6—C5	176.33 (16)
S1—N1—C7—C8	−178.80 (14)	C1—C6—C5—C4	0.2 (3)
O3—C7—C8—C13	−20.8 (3)	C6—C5—C4—C3	1.0 (3)
N1—C7—C8—C13	159.45 (19)	C6—C5—C4—Cl1	−178.71 (16)
O3—C7—C8—C9	158.7 (2)	C2—C3—C4—C5	−1.4 (3)
N1—C7—C8—C9	−21.0 (3)	C2—C3—C4—Cl1	178.32 (18)
C9—C8—C13—C12	0.7 (3)	C13—C8—C9—C10	−0.3 (3)
C7—C8—C13—C12	−179.7 (2)	C7—C8—C9—C10	−179.8 (2)
C4—C3—C2—C1	0.5 (3)	C8—C9—C10—C11	−0.6 (4)
C3—C2—C1—C6	0.7 (3)	C8—C13—C12—C11	−0.2 (3)
C3—C2—C1—S1	−176.74 (17)	C13—C12—C11—F1	179.7 (2)
O2—S1—C1—C2	−151.16 (17)	C13—C12—C11—C10	−0.8 (4)
O1—S1—C1—C2	−19.5 (2)	C9—C10—C11—F1	−179.3 (2)
N1—S1—C1—C2	91.82 (19)	C9—C10—C11—C12	1.2 (4)
O2—S1—C1—C6	31.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4	0.83 (3)	1.91 (3)	2.733 (3)	171 (2)
O4—H1*O*4···O1$^{\mathrm{i}}$	0.79 (3)	2.25 (3)	2.884 (2)	138 (3)
O4—H2*O*4···O2$^{\mathrm{ii}}$	0.82 (2)	2.29 (3)	2.955 (2)	139 (3)
O4—H2*O*4···O3$^{\mathrm{ii}}$	0.82 (2)	2.16 (3)	2.841 (2)	141 (3)
C5—H5···O1$^{\mathrm{iii}}$	0.93	2.54	3.124 (3)	121

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