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The crystal structures and Hirshfeld surface analysis of the 2-iodophenyl- and 4,5-difluoro-2-iodophenyl derivatives of benzenesulfonamide

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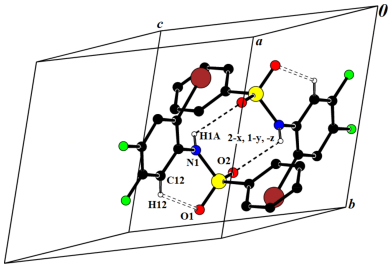
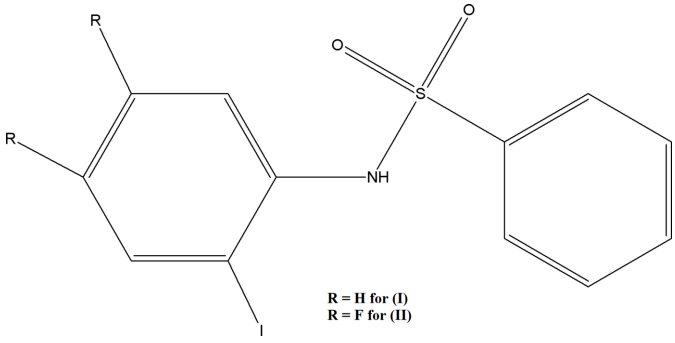
Two new benzenesulfonyl derivatives, *N*-(2-iodophenyl)benzenesulfonamide, $C_{12}H_{10}INO_2$, (**I**), and *N*-(4,5-difluoro-2-iodophenyl)benzenesulfonamide, $C_{12}H_8F_2INO_2S$, (**II**) were synthesized and structurally characterized. In both molecular structures, the conformation of the N—C bond in the –SO_2—NH—C segment is *gauche* relative to the S=O bond. For (**I**), the crystal packing is dominated by N—H···O hydrogen-bonding interactions that link the molecules into chains extending parallel to [010]. In the case of (**II**), the molecules are linked by N—H···O(S) hydrogen bonds into dimers that are located on centers of inversion. These findings are consistent with the results of Hirshfeld surface analyses.

1. Chemical context

Sulfonamide-containing compounds, often referred to as sulfa drugs, form a significant class of pharmacologically active agents. These molecules, which may incorporate one or more pharmacological scaffolds, demonstrate a broad spectrum of biological activities including antiviral, anticancer, antibacterial, anti-carbonic anhydrase (CA), diuretic, COX-2 inhibitory, and protease inhibitory effects (Madhan *et al.*, 2024*a*,*b*,*c*). The sulfonamide moiety is recognized as an important structural unit in medicinal chemistry and is present in many widely marketed drugs (Supuran, 2003; Elgemeie *et al.*, 2019). Since their discovery, sulfonamides have been extensively used as antibiotics (Zhao *et al.*, 2016), particularly for treating infections like malaria, tuberculosis, or HIV, by targeting the dihydropteroate synthase (DHPS) pathway (Dennis *et al.*, 2018). Even after the advent of penicillin, sulfa drugs have retained their relevance in clinical settings due to their diverse therapeutic actions, including antitumor, anticancer, and antithyroid activities (Scozzafava *et al.*, 2003). Various sulfonamide derivatives serve as chemotherapeutic agents, exhibiting antibacterial, antifungal, antitumor, and hypoglycemic properties (Chohan *et al.*, 2010; El-Sayed *et al.*, 2011 Seri *et al.*, 2000). Benzenesulfonamide derivatives are particularly known for their antitumor and antifungal activities. Crystallographic studies of these compounds reveal structural parameters consistent with other sulfonamide-based molecules (Chakkaravarthi *et al.*, 2007; Li & Yang, 2006). Continued interest in sulfonamides stems from their enduring role in treating bacterial infections, their chemical versatility, and their effectiveness despite the rise of newer antibiotic classes. Modern synthetic approaches aim to produce sulfon-



amide-functionalized heterocycles with enhanced antiviral and antimicrobial profiles (Madhan *et al.*, 2024*a*). Research into N-sulfonylated I and F atom-substituted compounds is motivated by the observed enhancement of biological activity. Hence, the introduction of fluorine atoms into drugs is increasingly common due to the strong electron-withdrawing character and small atomic radius of the fluorine atom, which significantly influences the physiological, pharmacological and metabolic properties of a compound (Mueller *et al.*, 2007; Purser *et al.*, 2008). The availability of multiple aromatic groups in N-sulfonylated 2-iodophenyl imposes also the possibility for versatile stacking patterns, which may be competitive to the conventional hydrogen-bonding interactions in the crystal packing.



In the context given above, we report herein the crystal structure determinations and Hirshfeld surface analyses of two new 2-iodophenyl benzenesulfonamides: *N*-(2-iodophenyl)benzenesulfonamide, $C_{12}H_{10}INO_2$, (**I**), and *N*-(4,5-difluoro-2-iodophenyl)benzenesulfonamide, $C_{12}H_8F_2INO_2S$, (**II**), which feature a complex interplay of weak hydrogen bonding and π–π interactions.

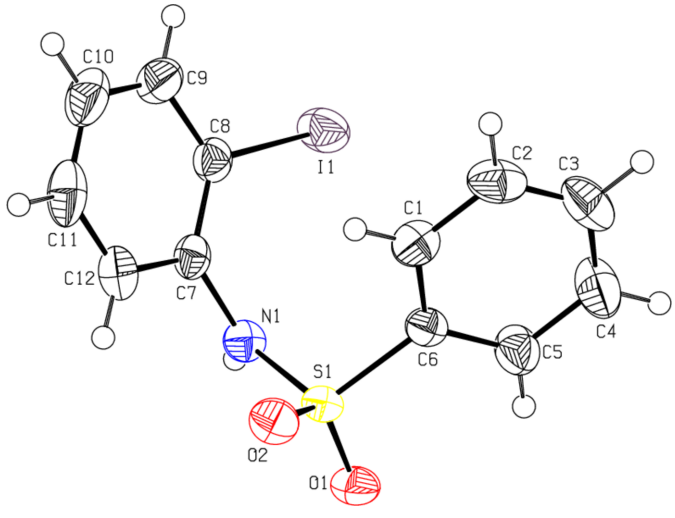


Figure 1
The molecular structure of compound (**I**), with atom labeling and displacement ellipsoids drawn at the 50% probability level.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.37	3.144 (3)	149
C3—H3···O1^{ii}	0.93	2.57	3.352 (4)	142

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

2. Structural commentary

The molecular structures of (**I**) and (**II**) are shown in Figs. 1 and 2, respectively. In both cases, the conformation of the N—C bond in the –SO_2—NH—C segment is *gauche* relative to the S=O bond. The molecule is twisted at the S—N bond with a torsion angle of C7—N1—S1—C6 = −69.0 (2)° for (**I**) and −61.1 (6)° for (**II**) compared to the values of −72.83 (15) and 61.9 (3)° in *N*-(phenyl)-2-nitrobenzenesulfonamide (Chaithanya *et al.*, 2012*a*) and 4-nitro-*N*-phenylbenzenesulfonamide (Chaithanya *et al.*, 2012*b*), respectively. The two benzene rings are tilted relative to each other by 44.1 (1)° for (**I**) and 73.1 (1)° for (**II**). The molecular configuration of (**II**) is stabilized by a weak intramolecular hydrogen bond C12—H12···O1 (Table 1) with one of the sulfone O-atoms as acceptor, which generates an *S*(6) ring motif. Other structural parameters (bond lengths and angles) in the molecules of (**I**) and (**II**) agree well with those reported for related compounds (Madhan *et al.*, 2022, 2023*a*,*b*, 2024*a*,*b*,*c*).

3. Supramolecular features

In the crystal structure of (**I**), intermolecular N—H···O hydrogen-bonding interactions (Table 1) link the molecules into *C*(4) chains (Etter *et al.*, 1990) running parallel to [010]

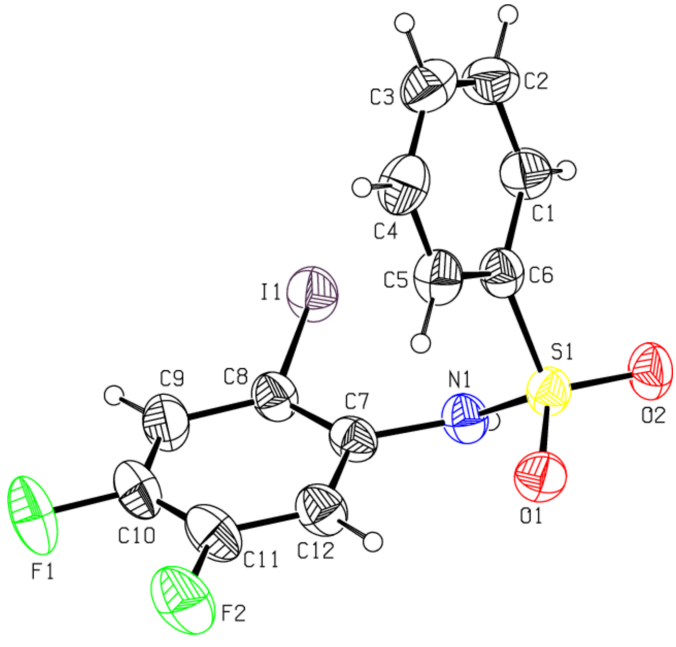


Figure 2
The molecular structure of compound (**II**), with atom labeling and displacement ellipsoids drawn at the 50% probability level.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.86	2.58	3.169 (8)	126
C12—H12···O1	0.93	2.27	2.916 (11)	126

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

while C—H···O interactions interlink these chains (Fig. 3). In addition, π–π interactions are present with a centroid-to-centroid distance $Cg2\cdots Cg2\ (2-x, 1-y, 1-z) = 3.747\ (2)$ Å and a slippage of 1.035 Å ($Cg2$ is the centroid of phenyl ring C7–C1).

In the crystal structure of (**II**), molecules are linked by N—H···O(S) hydrogen-bonding interactions (Table 2, Fig. 4) into inversion-related dimers with an $R_2^2(8)$ graph-set motif (Etter *et al.*, 1990). Like for (**I**), π–π interactions are present

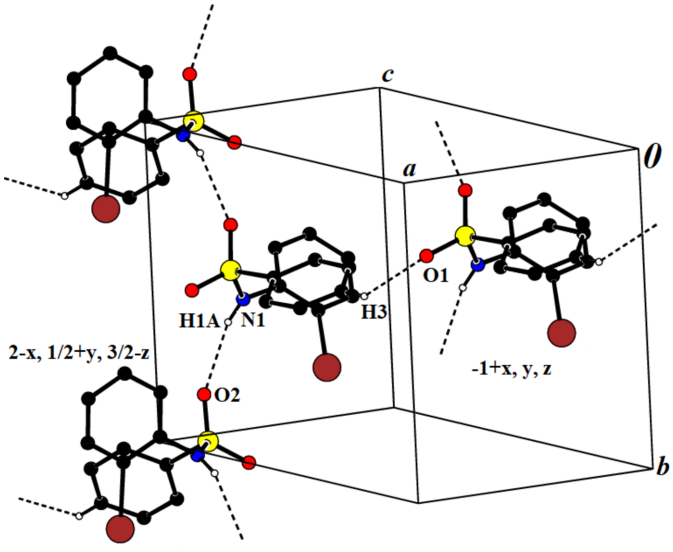


Figure 3
Crystal packing of compound (**I**), showing the N—H···O and C—H···O interaction that link the molecules into chains.

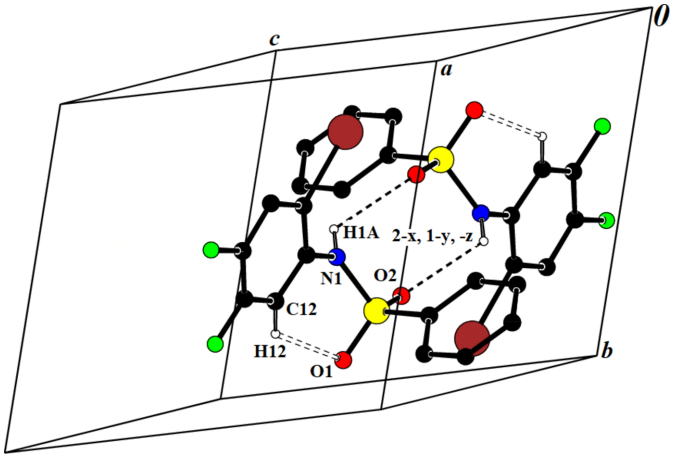


Figure 4
Crystal packing of compound (**II**), showing the N—H···O(S) hydrogen-bonding interactions that lead to inversion-related dimers.

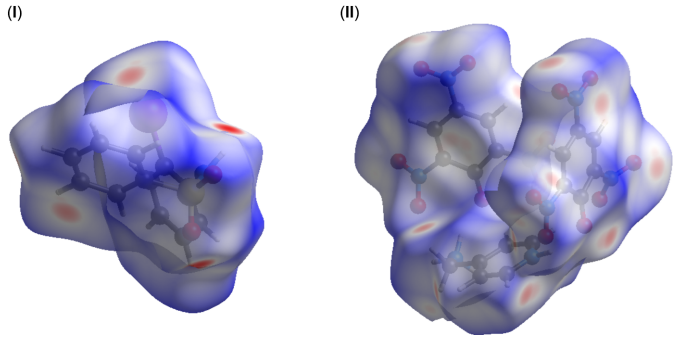


Figure 5
The Hirshfeld surfaces of compounds (**I**) and (**II**) mapped over d_{norm}.

that consolidate the crystal packing, here with centroid-to-centroid distances $Cg1\cdots Cg1(1-x, 2-y, -z) = 3.621\ (2)$ and a slippage of 0.998 Å and $Cg2\cdots Cg2(2-x, 1-y, 1-z) = 3.797\ (2)$ Å and a slippage of 1.617 Å ($Cg1$ and $Cg2$ are the centroids of the C1–C6 and C7–C12 rings, respectively).

4. Hirshfeld surface analysis

In order to quantify the intermolecular interactions in the crystals of (**I**) and (**II**), Hirshfeld surfaces and two-dimensional fingerprint plots were generated using *CrystalExplorer* (Spackman *et al.*, 2021).

Plots of d_{norm} use the normalized functions d_i and d_e (Fig. 5), with white surfaces indicating contacts with distances equal to the sum of van der Waals (vdW) radii, while red and blue colors reflect contacts at the distances below and above sum of the corresponding vdW radii, respectively. Two-dimensional fingerprint plots showing the occurrence of all intermolecular contacts (McKinnon *et al.*, 2007) and are presented in Fig. 6. H···H in (**I**) and O···H/H···O contacts in (**II**) represent the largest contributions to the Hirshfeld surfaces (37.4 and 44.7%, respectively). Beyond these largest fractions, short contacts are O···H/H···O (21.7%) for (**I**) and O···C/C···O (17.2%) for (**II**) (Fig. 6*c*), C···H/H···C (16.5%) for (**I**) and O···O (11%) for (**II**) (Fig. 6*d*). The significant increase in the O···H/H···O contributions when moving from (**I**) to (**II**) reflects growing significance of C—H···O binding. This is in line with a larger number of the available intermolecular O-atom acceptors in the latter case. Accordingly, a pair of spikes identifying O···H/H···O contacts on the plots in the case of (**I**) is more diffuse.

In brief, the Hirshfeld surface analyses complement the main merit of the structure analyses, and together they suggest possibilities for controlling the supramolecular behavior of benzenesulfonamide as possible biomedical materials.

5. Database survey

A search of the Cambridge Structural Database (CSD, version 5.37; Groom *et al.*, 2016) indicated 123 compounds incorporating the phenylsulfonamide moiety. The bond lengths and angles in (**I**) and (**II**) are very close to those observed in 2,4-dimethyl-*N*-(phenyl)benzenesulfonamide (Gowda *et al.*,

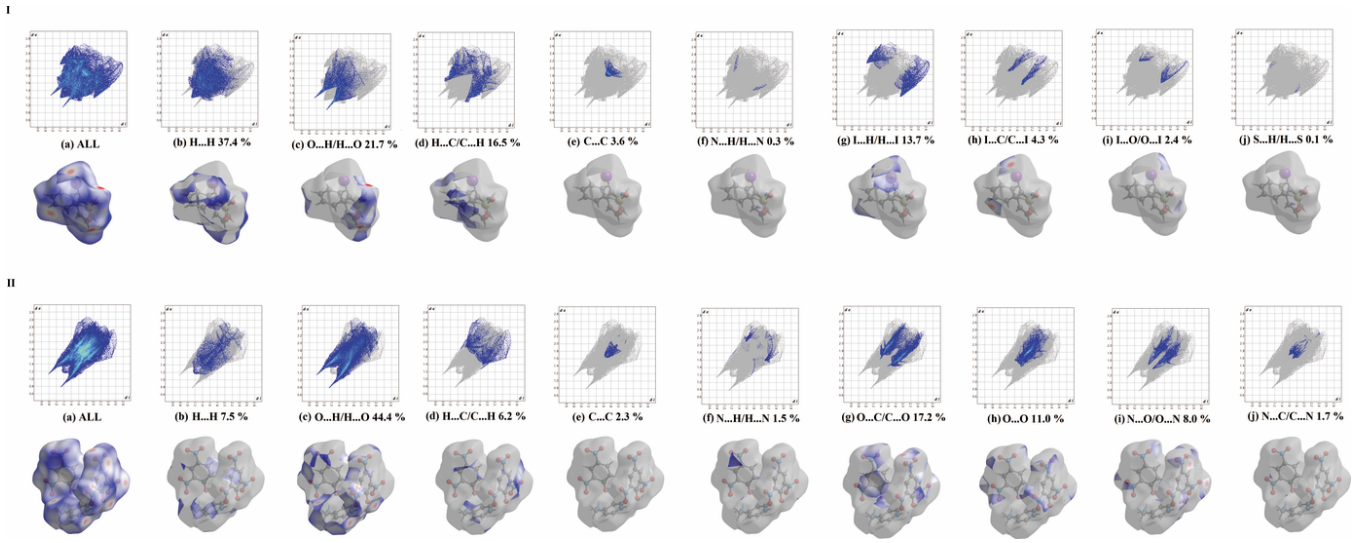


Figure 6
Two-dimensional fingerprint plots for (**I**) and delineated into the principal contributions of H···H, O···H/H···O, C···H/H···C, I···H/H···I, I···C/C···I, C···C and I···O/O···I contacts and for (**II**) O···H/H···O, O···C/C···O, O···O, N···O/O···N, H···H, H···C/C···H, C···C, N···C/C···N and N···H/H···N. Other contributors account for less than 1.0% contacts to the surface areas.

2009*a*), 4-chloro-2-methyl-*N*-(phenyl)benzenesulfonamide (Gowda *et al.*, 2009*b*), 4-methyl-*N*-(3,4-dimethylphenyl) benzenesulfonamide (Gowda *et al.*, 2009*c*) and other aryl sulfonamides Perlovich *et al.*, 2006; Tatsuta *et al.*, 2009; Arora & Sundaralingam, 1971; Gelbrich *et al.*, 2007). Of these, the most closely related examples are provided by structures of bromosubstituted 3-methyl-1-(phenylsulfonyl)-1*H*-indole derivatives (Madhan *et al.*, 2024*b*).

6. Synthesis and crystallization

(**I**): To a solution of 2-iodoaniline (2 g, 9.17 mmol) in dry dichloromethane (DCM; 10 ml), benzenesulfonyl chloride (1.42 ml, 11.01 mmol) and pyridine (1.11 ml, 13.76 mmol) were slowly added and stirred at room temperature for 8 h under nitrogen atmosphere. After completion of the reaction (monitored by TLC), it was poured into ice water containing conc. HCl (1 ml), extracted with DCM (3 × 10 ml) then washed with water (2 × 20 ml) and dried (Na_2SO_4). Removal of the solvent *in vacuo* followed by trituration of the crude product with diethyl ether (5 ml) afforded (**I**) (2.43 g, 84%) as a colorless solid. M.p: 363–365 K. ^{1}H-NMR (300 MHZ, $CDCl_3$): δ 7.70 (*d*, *J* = 7.5 Hz, 2H), 7.61 (*t*, *J* = 7.5 Hz, 2H), 7.55–7.49 (*m*, 1 H), 7.41–7.36 (*m*, 2H), 7.28 (*t*, *J* = 7.5 Hz, 1H), 6.82–6.77 (*m*, 2H) ppm; 13C{1H}-NMR (75 MHz, $CDCl_3$): δ 139.1, 138.8, 137.4, 133.3, 129.6, 127.4, 122.9, 92.5 ppm.

(**II**): To a solution of 4,5-difluoro2-iodoaniline (2 g, 7.84 mmol) in dry DCM (10 ml), benzenesulfonyl chloride (1.21 ml, 9.41 mmol) and pyridine (0.95 ml, 11.76 mmol) were slowly added and stirred at room temperature for 8 h under nitrogen atmosphere. After completion of the reaction (monitored by TLC), it was poured into ice water containing conc. HCl (1 ml), extracted with DCM (3 × 10 ml) then washed with water (2 × 20 ml) and dried (Na_2SO_4). Removal of solvent *in vacuo* followed by trituration of the crude product with diethyl ether (5 mL) afforded benzenesulfonamide (**II**) (2.56 g, 83%) as a colorless solid. M.p: 393–395 K. ^{1}H-NMR (300 MHZ, $CDCl_3$): δ 7.58 (*d*, *J* = 7.5 Hz, 2H), 7.45–7.39 (*m*, 2 H), 7.30–7.26 (*m*, 3 H)ppm; 13C{1H}-NMR (75 MHz, $CDCl_3$): δ 150.7 (*dd*, $^1J_{C-F}$ = 249.4 Hz, $^2J_{C-F}$ = 12.7 Hz), 147.8 (*dd*, $^1J_{C-F}$ = 252 Hz, $^2J_{C-F}$ = 12.7 Hz), 138.4, 134.3 (*dd*, $^1J_{C-F}$ = 82 Hz, $^2J_{C-F}$ = 3 Hz), 133.7, 129.3, 127.4, 126.8 (*d*, J_{C-F} = 19.5 Hz), 112.3 (*d*, J_{C-F} = 21.7 Hz) ppm.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All hydrogen atoms were positioned geometrically and refined as riding with N—H = 0.86 and C—H = 0.93 Å (aromatic CH) with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other H atoms.

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Table 3
Experimental details.

	(**I**)	(**II**)
Crystal data		
Chemical formula	$C_{12}H_{10}INO_2S$	$C_{12}H_8F_2INO_2S$
M_r	359.17	395.15
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	298	298
a, b, c (Å)	8.3648 (4), 9.8537 (4), 15.3923 (8)	8.2688 (5), 8.5178 (5), 10.4329 (7)
α, β, γ (°)	90, 90.955 (2), 90	81.291 (2), 80.503 (2), 68.383 (2)
V (Å^3)	1268.52 (10)	670.47 (7)
Z	4	2
Radiation type	Mo $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	2.68	20.44
Crystal size (mm)	0.35 × 0.25 × 0.08	0.22 × 0.10 × 0.05
Data collection		
Diffractometer	Bruker D8 Venture Diffractometer	Bruker D8 Venture Diffractometer
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.672, 0.971	0.240, 0.521
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29284, 2591, 2386	14553, 2476, 2367
R_{int}	0.049	0.062
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.625	0.605
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.069, 1.09	0.072, 0.203, 1.14
No. of reflections	2591	2476
No. of parameters	155	173
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.54, −0.56	1.66, −0.95

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2020), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

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The crystal structures and Hirshfeld surface analysis of the 2-iodophenyl- and 4,5-difluoro-2-iodophenyl derivatives of benzenesulfonamide

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Computing details

N-(2-Iodophenyl)benzenesulfonamide (I)

Crystal data

$C_{12}H_{10}INO_2S$
$M_r = 359.17$
Monoclinic, $P2_1/c$
$a = 8.3648$ (4) Å
$b = 9.8537$ (4) Å
$c = 15.3923$ (8) Å
$\beta = 90.955$ (2)°
$V = 1268.52$ (10) Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.881$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 29284 reflections
$\theta = 1.4$–25.0°
$\mu = 2.68$ mm^{-1}
$T = 298$ K
Prism, colourless
0.35 × 0.25 × 0.08 mm

Data collection

Bruker D8 Venture Diffractometer
Radiation source: micro focus sealed tube
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min} = 0.672$, $T_{max} = 0.971$
29284 measured reflections
2591 independent reflections
2386 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.4°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.069$
$S = 1.09$
2591 reflections
155 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 1.2298P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}
Extinction correction: SHELXL-2019/2 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0085 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5786 (3)	0.4285 (3)	0.74857 (19)	0.0393 (6)
H1	0.601455	0.365298	0.705621	0.047*
C2	0.4228 (4)	0.4717 (4)	0.7611 (2)	0.0488 (8)
H2	0.340905	0.439224	0.725207	0.059*
C3	0.3886 (4)	0.5624 (4)	0.8263 (3)	0.0539 (9)
H3	0.283767	0.590431	0.834558	0.065*
C4	0.5092 (4)	0.6113 (4)	0.8791 (3)	0.0576 (9)
H4	0.485310	0.671374	0.923622	0.069*
C5	0.6662 (4)	0.5720 (3)	0.8666 (2)	0.0451 (7)
H5	0.747984	0.606424	0.901827	0.054*
C6	0.6996 (3)	0.4810 (3)	0.80115 (17)	0.0310 (5)
C7	0.8849 (3)	0.4506 (3)	0.61007 (17)	0.0311 (5)
C8	0.7791 (3)	0.5277 (3)	0.55998 (18)	0.0342 (6)
C9	0.7163 (4)	0.4759 (4)	0.4824 (2)	0.0481 (8)
H9	0.646575	0.528401	0.448826	0.058*
C10	0.7567 (4)	0.3479 (4)	0.4552 (2)	0.0547 (9)
H10	0.713859	0.313449	0.403539	0.066*
C11	0.8612 (5)	0.2705 (4)	0.5049 (2)	0.0537 (9)
H11	0.888068	0.183527	0.486772	0.064*
C12	0.9263 (4)	0.3215 (3)	0.5814 (2)	0.0420 (7)
H12	0.998086	0.269303	0.613894	0.050*
N1	0.9533 (3)	0.4996 (2)	0.69018 (14)	0.0316 (5)
H1A	1.022622	0.564040	0.689506	0.038*
O1	0.9978 (2)	0.4978 (2)	0.84793 (13)	0.0405 (5)
O2	0.9031 (3)	0.28915 (19)	0.77277 (14)	0.0408 (5)
S1	0.90001 (7)	0.43340 (6)	0.78303 (4)	0.02932 (16)
I1	0.71045 (2)	0.72190 (2)	0.59939 (2)	0.04598 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0361 (15)	0.0437 (16)	0.0381 (15)	−0.0119 (12)	0.0004 (12)	0.0013 (13)
C2	0.0328 (15)	0.059 (2)	0.0542 (19)	−0.0143 (14)	−0.0079 (13)	0.0148 (16)
C3	0.0307 (15)	0.055 (2)	0.076 (2)	0.0046 (14)	0.0086 (15)	0.0110 (18)
C4	0.0435 (18)	0.057 (2)	0.072 (2)	0.0047 (16)	0.0105 (17)	−0.0175 (19)
C5	0.0360 (15)	0.0491 (18)	0.0500 (17)	−0.0010 (13)	0.0013 (13)	−0.0111 (15)
C6	0.0283 (12)	0.0328 (13)	0.0320 (13)	−0.0013 (10)	0.0003 (10)	0.0059 (11)
C7	0.0291 (12)	0.0344 (14)	0.0300 (13)	−0.0044 (10)	0.0080 (10)	−0.0005 (11)
C8	0.0319 (13)	0.0387 (15)	0.0320 (13)	−0.0018 (11)	0.0063 (11)	0.0002 (11)

C9	0.0418 (16)	0.068 (2)	0.0343 (15)	−0.0020 (15)	−0.0015 (12)	−0.0053 (15)
C10	0.053 (2)	0.069 (2)	0.0419 (17)	−0.0123 (18)	0.0080 (15)	−0.0218 (17)
C11	0.060 (2)	0.0462 (19)	0.056 (2)	−0.0063 (15)	0.0219 (17)	−0.0208 (16)
C12	0.0412 (16)	0.0392 (15)	0.0461 (16)	0.0024 (13)	0.0133 (13)	−0.0029 (13)
N1	0.0285 (11)	0.0322 (11)	0.0340 (11)	−0.0050 (9)	0.0015 (9)	0.0027 (9)
O1	0.0329 (10)	0.0483 (12)	0.0399 (11)	−0.0002 (9)	−0.0090 (8)	0.0000 (9)
O2	0.0485 (12)	0.0293 (10)	0.0445 (11)	0.0047 (8)	−0.0005 (9)	0.0073 (8)
S1	0.0274 (3)	0.0295 (3)	0.0309 (3)	0.0011 (2)	−0.0029 (2)	0.0029 (3)
I1	0.04805 (15)	0.03807 (15)	0.05150 (16)	0.00919 (8)	−0.00858 (9)	0.00039 (8)

Geometric parameters (Å, º)

C1—C6	1.385 (4)	C7—N1	1.434 (3)
C1—C2	1.388 (5)	C8—C9	1.393 (4)
C1—H1	0.9300	C8—I1	2.091 (3)
C2—C3	1.377 (5)	C9—C10	1.373 (5)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.372 (5)	C10—C11	1.382 (6)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.386 (5)	C11—C12	1.384 (5)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.380 (4)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.640 (2)
C6—S1	1.768 (3)	N1—H1A	0.8600
C7—C8	1.390 (4)	O1—S1	1.429 (2)
C7—C12	1.392 (4)	O2—S1	1.430 (2)
C6—C1—C2	118.8 (3)	C9—C8—I1	118.9 (2)
C6—C1—H1	120.6	C10—C9—C8	120.5 (3)
C2—C1—H1	120.6	C10—C9—H9	119.8
C3—C2—C1	120.5 (3)	C8—C9—H9	119.8
C3—C2—H2	119.7	C9—C10—C11	119.6 (3)
C1—C2—H2	119.7	C9—C10—H10	120.2
C4—C3—C2	120.0 (3)	C11—C10—H10	120.2
C4—C3—H3	120.0	C10—C11—C12	120.5 (3)
C2—C3—H3	120.0	C10—C11—H11	119.8
C3—C4—C5	120.6 (3)	C12—C11—H11	119.8
C3—C4—H4	119.7	C11—C12—C7	120.3 (3)
C5—C4—H4	119.7	C11—C12—H12	119.8
C6—C5—C4	119.1 (3)	C7—C12—H12	119.8
C6—C5—H5	120.5	C7—N1—S1	120.34 (18)
C4—C5—H5	120.5	C7—N1—H1A	119.8
C5—C6—C1	121.0 (3)	S1—N1—H1A	119.8
C5—C6—S1	119.4 (2)	O1—S1—O2	120.50 (13)
C1—C6—S1	119.6 (2)	O1—S1—N1	105.77 (12)
C8—C7—C12	118.9 (3)	O2—S1—N1	107.07 (13)
C8—C7—N1	122.3 (2)	O1—S1—C6	107.76 (13)
C12—C7—N1	118.8 (3)	O2—S1—C6	107.45 (13)

C7—C8—C9	120.2 (3)	N1—S1—C6	107.72 (12)
C7—C8—I1	121.0 (2)		
C6—C1—C2—C3	−1.8 (5)	C9—C10—C11—C12	−0.5 (5)
C1—C2—C3—C4	0.4 (5)	C10—C11—C12—C7	1.2 (5)
C2—C3—C4—C5	1.0 (6)	C8—C7—C12—C11	−0.8 (4)
C3—C4—C5—C6	−1.1 (6)	N1—C7—C12—C11	179.3 (3)
C4—C5—C6—C1	−0.4 (5)	C8—C7—N1—S1	110.1 (3)
C4—C5—C6—S1	178.5 (3)	C12—C7—N1—S1	−70.0 (3)
C2—C1—C6—C5	1.8 (4)	C7—N1—S1—O1	176.0 (2)
C2—C1—C6—S1	−177.1 (2)	C7—N1—S1—O2	46.3 (2)
C12—C7—C8—C9	−0.1 (4)	C7—N1—S1—C6	−69.0 (2)
N1—C7—C8—C9	179.7 (3)	C5—C6—S1—O1	3.3 (3)
C12—C7—C8—I1	179.6 (2)	C1—C6—S1—O1	−177.9 (2)
N1—C7—C8—I1	−0.6 (3)	C5—C6—S1—O2	134.5 (2)
C7—C8—C9—C10	0.8 (5)	C1—C6—S1—O2	−46.6 (3)
I1—C8—C9—C10	−178.9 (3)	C5—C6—S1—N1	−110.4 (2)
C8—C9—C10—C11	−0.5 (5)	C1—C6—S1—N1	68.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.37	3.144 (3)	149
C3—H3···O1^{ii}	0.93	2.57	3.352 (4)	142

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

N-(4,5-Difluoro-2-iodophenyl)benzenesulfonamide (II)

Crystal data

$C_{12}H_8F_2INO_2S$
M_r = 395.15
Triclinic, $P\overline{1}$
a = 8.2688 (5) Å
b = 8.5178 (5) Å
c = 10.4329 (7) Å
α = 81.291 (2)°
β = 80.503 (2)°
γ = 68.383 (2)°
V = 670.47 (7) Å^3
Z = 2
F(000) = 380
D_x = 1.957 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 14553 reflections
θ = 1.4–25.0°
μ = 20.44 mm^{-1}
T = 298 K
Prism, colourless
0.22 × 0.10 × 0.05 mm

Data collection

Bruker D8 Venture Diffractometer
Radiation source: micro focus sealed tube
ω and *φ* scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.240, T_{max} = 0.521
14553 measured reflections
2476 independent reflections
2367 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 69.0°, θ_{min} = 4.3°
h = −9→10
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.203$
$S = 1.14$
2476 reflections
173 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1461P)^2 + 0.5316P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.66$ e Å^{-3}
$\Delta\rho_{min} = -0.94$ e Å^{-3}
Extinction correction: SHELXL-2019/2 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6043 (11)	0.6893 (10)	0.0209 (8)	0.0690 (17)
H1	0.692052	0.606880	−0.025462	0.083*
C2	0.4337 (12)	0.7289 (12)	0.0029 (10)	0.079 (2)
H2	0.405262	0.675110	−0.056593	0.095*
C3	0.3033 (11)	0.8488 (11)	0.0732 (10)	0.081 (2)
H3	0.186655	0.875898	0.061633	0.097*
C4	0.3467 (10)	0.9284 (11)	0.1607 (10)	0.080 (2)
H4	0.257809	1.008690	0.208075	0.096*
C5	0.5138 (10)	0.8932 (9)	0.1794 (8)	0.0674 (16)
H5	0.540488	0.948502	0.238810	0.081*
C6	0.6479 (9)	0.7712 (8)	0.1076 (6)	0.0582 (14)
C7	0.8376 (8)	0.5366 (8)	0.3596 (6)	0.0571 (14)
C8	0.7924 (10)	0.3964 (9)	0.4144 (7)	0.0614 (15)
C9	0.7196 (12)	0.3840 (12)	0.5445 (8)	0.078 (2)
H9	0.692000	0.289102	0.581056	0.093*
C10	0.6899 (13)	0.5163 (13)	0.6173 (8)	0.081 (2)
C11	0.7353 (14)	0.6529 (12)	0.5661 (9)	0.082 (2)
C12	0.8116 (11)	0.6642 (10)	0.4384 (8)	0.0700 (18)
H12	0.845408	0.756575	0.405198	0.084*
N1	0.9216 (7)	0.5461 (7)	0.2298 (6)	0.0597 (12)
H1A	1.004065	0.457002	0.202938	0.072*
O1	0.8802 (7)	0.8532 (7)	0.1911 (6)	0.0691 (12)
O2	0.9746 (7)	0.6771 (7)	0.0082 (5)	0.0717 (13)
S1	0.8673 (2)	0.7215 (2)	0.12842 (16)	0.0582 (5)
I1	0.83499 (6)	0.19041 (6)	0.30905 (4)	0.0749 (4)
F1	0.6177 (10)	0.5090 (9)	0.7433 (5)	0.116 (2)
F2	0.7077 (11)	0.7780 (9)	0.6410 (6)	0.112 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.085 (5)	0.061 (4)	0.068 (4)	−0.032 (3)	−0.006 (3)	−0.014 (3)
C2	0.089 (5)	0.080 (5)	0.083 (5)	−0.043 (4)	−0.024 (4)	−0.005 (4)
C3	0.064 (4)	0.076 (5)	0.102 (6)	−0.028 (4)	−0.017 (4)	0.007 (4)
C4	0.061 (4)	0.074 (5)	0.091 (6)	−0.016 (3)	0.008 (4)	−0.010 (4)
C5	0.074 (4)	0.057 (3)	0.068 (4)	−0.020 (3)	0.004 (3)	−0.014 (3)
C6	0.067 (3)	0.054 (3)	0.054 (3)	−0.023 (3)	−0.001 (3)	−0.007 (3)
C7	0.056 (3)	0.059 (3)	0.053 (3)	−0.012 (2)	−0.007 (2)	−0.015 (3)
C8	0.070 (4)	0.061 (4)	0.053 (3)	−0.022 (3)	−0.004 (3)	−0.010 (3)
C9	0.092 (5)	0.084 (5)	0.059 (4)	−0.038 (4)	0.002 (4)	−0.006 (4)
C10	0.095 (5)	0.092 (6)	0.048 (4)	−0.026 (4)	0.001 (3)	−0.014 (4)
C11	0.100 (6)	0.078 (5)	0.060 (4)	−0.015 (4)	−0.011 (4)	−0.023 (4)
C12	0.085 (5)	0.062 (4)	0.063 (4)	−0.024 (3)	−0.008 (3)	−0.014 (3)
N1	0.065 (3)	0.056 (3)	0.055 (3)	−0.018 (2)	0.000 (2)	−0.014 (2)
O1	0.079 (3)	0.064 (3)	0.073 (3)	−0.033 (2)	−0.002 (2)	−0.020 (2)
O2	0.078 (3)	0.076 (3)	0.059 (3)	−0.030 (3)	0.014 (2)	−0.018 (2)
S1	0.0632 (9)	0.0577 (9)	0.0560 (9)	−0.0254 (7)	0.0028 (7)	−0.0127 (7)
I1	0.0903 (5)	0.0698 (5)	0.0695 (5)	−0.0344 (3)	0.0010 (3)	−0.0166 (3)
F1	0.154 (6)	0.122 (5)	0.052 (3)	−0.036 (4)	0.022 (3)	−0.018 (3)
F2	0.156 (6)	0.104 (4)	0.077 (3)	−0.038 (4)	−0.001 (3)	−0.045 (3)

Geometric parameters (Å, º)

C1—C2	1.363 (12)	C7—N1	1.422 (9)
C1—C6	1.386 (10)	C8—C9	1.396 (10)
C1—H1	0.9300	C8—I1	2.100 (7)
C2—C3	1.377 (14)	C9—C10	1.378 (13)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.376 (14)	C10—F1	1.353 (9)
C3—H3	0.9300	C10—C11	1.360 (15)
C4—C5	1.343 (12)	C11—F2	1.346 (10)
C4—H4	0.9300	C11—C12	1.382 (12)
C5—C6	1.408 (10)	C12—H12	0.9300
C5—H5	0.9300	N1—S1	1.655 (6)
C6—S1	1.748 (7)	N1—H1A	0.8600
C7—C12	1.394 (10)	O1—S1	1.425 (5)
C7—C8	1.394 (10)	O2—S1	1.425 (5)
C2—C1—C6	120.4 (8)	C9—C8—I1	116.3 (6)
C2—C1—H1	119.8	C10—C9—C8	118.2 (8)
C6—C1—H1	119.8	C10—C9—H9	120.9
C1—C2—C3	119.9 (8)	C8—C9—H9	120.9
C1—C2—H2	120.1	F1—C10—C11	118.9 (9)
C3—C2—H2	120.1	F1—C10—C9	119.6 (9)
C4—C3—C2	119.7 (7)	C11—C10—C9	121.4 (8)
C4—C3—H3	120.2	F2—C11—C10	119.7 (8)

C2—C3—H3	120.2	F2—C11—C12	119.5 (9)
C5—C4—C3	121.8 (8)	C10—C11—C12	120.8 (8)
C5—C4—H4	119.1	C11—C12—C7	119.7 (8)
C3—C4—H4	119.1	C11—C12—H12	120.1
C4—C5—C6	118.9 (8)	C7—C12—H12	120.1
C4—C5—H5	120.5	C7—N1—S1	122.7 (4)
C6—C5—H5	120.5	C7—N1—H1A	118.7
C1—C6—C5	119.3 (7)	S1—N1—H1A	118.7
C1—C6—S1	120.1 (6)	O1—S1—O2	118.7 (4)
C5—C6—S1	120.6 (5)	O1—S1—N1	107.5 (3)
C12—C7—C8	118.5 (7)	O2—S1—N1	105.5 (3)
C12—C7—N1	119.4 (7)	O1—S1—C6	108.4 (3)
C8—C7—N1	121.8 (6)	O2—S1—C6	109.5 (3)
C7—C8—C9	121.3 (7)	N1—S1—C6	106.5 (3)
C7—C8—I1	122.4 (5)		

C6—C1—C2—C3	−1.2 (12)	F1—C10—C11—C12	179.1 (9)
C1—C2—C3—C4	0.3 (13)	C9—C10—C11—C12	0.7 (15)
C2—C3—C4—C5	0.4 (14)	F2—C11—C12—C7	−178.8 (8)
C3—C4—C5—C6	−0.2 (13)	C10—C11—C12—C7	2.0 (13)
C2—C1—C6—C5	1.4 (11)	C8—C7—C12—C11	−2.8 (11)
C2—C1—C6—S1	−179.2 (6)	N1—C7—C12—C11	−177.8 (7)
C4—C5—C6—C1	−0.7 (11)	C12—C7—N1—S1	−47.0 (8)
C4—C5—C6—S1	179.9 (6)	C8—C7—N1—S1	138.2 (6)
C12—C7—C8—C9	1.2 (11)	C7—N1—S1—O1	55.0 (6)
N1—C7—C8—C9	176.1 (7)	C7—N1—S1—O2	−177.4 (5)
C12—C7—C8—I1	−177.3 (5)	C7—N1—S1—C6	−61.1 (6)
N1—C7—C8—I1	−2.4 (9)	C1—C6—S1—O1	163.4 (6)
C7—C8—C9—C10	1.3 (13)	C5—C6—S1—O1	−17.1 (7)
I1—C8—C9—C10	179.9 (7)	C1—C6—S1—O2	32.5 (7)
C8—C9—C10—F1	179.3 (8)	C5—C6—S1—O2	−148.0 (6)
C8—C9—C10—C11	−2.3 (14)	C1—C6—S1—N1	−81.1 (6)
F1—C10—C11—F2	−0.1 (15)	C5—C6—S1—N1	98.4 (6)
C9—C10—C11—F2	−178.6 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.58	3.169 (8)	126
C12—H12···O1	0.93	2.27	2.916 (11)	126

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

*Geometry of stacking interactions for **I** and **II** (Å, °).*

Compound	Group A	Group B	Shortest contacts	CgA···CgB	Plane···CgB	ipa	sa
I	(C7–C12)	(C7–C12)^{ii}	3.747 (2)	3.747 (2)	3.602 (2)	0	16.0 (1)

II	(C1–C6)	(C1–C6)^{iii}	3.225 (2)	3.621 (2)	3.480 (2)	0	16.0 (2)
	(C7–C12)	(C7–C12)^{iv}	3.499 (2)	3.797 (2)	3.436 (2)	0	25.2 (2)

a) Cg is a group centroid; Plane···CgB is the distance between mean plane of Group A and centroid of the interacting Group B; ipa is interplanar angle; sa is slippage angle, which is the angle of CgA···CgB axis to the Group A mean plane normal. [Symmetry codes for **I**: (ii) 2-X,-Y,-Z; for **II**: (iii) 1-X,2-Y,-Z; (iv) 2-X,1-Y,1-Z.]