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[1-(4-Chloro-2-fluorophenylsulfonyl)-piperidin-4-yl]diphenylmethanol

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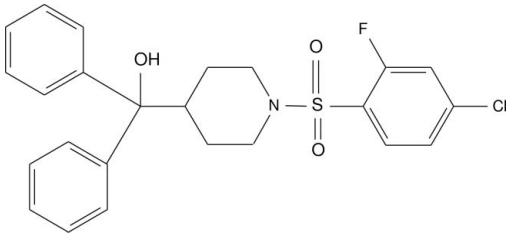
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.152; data-to-parameter ratio = 12.8.

In the title compound, $C_{24}H_{23}ClFNO_3S$, the piperidine ring is in a chair conformation. The geometry around the S atom is distorted tetrahedral. The dihedral angle between the least-squares plane, $P1$, defined by four C atoms of the piperidine ring, and the dihalo-substituted benzene ring is 49.80 (1)°. The dihedral angles between $P1$ and the two phenyl rings are 59.34 (1) and 73.81 (1)°. The two phenyl rings make a dihedral angle of 65.13 (14)°. The structure exhibits intermolecular hydrogen bonds of the types O—H···O and C—H···O.

Related literature

For related literature, see: Cremer & Pople (1975); Henderson *et al.* (1996); Li *et al.* (2006); Mao *et al.* (1998); Sugimoto *et al.* (1990).



Experimental

Crystal data

$C_{24}H_{23}ClFNO_3S$
M_r = 459.94
Monoclinic, $P2_1/c$
a = 10.238 (7) Å
b = 11.295 (4) Å
c = 21.072 (12) Å
β = 115.481 (2)°
V = 2200 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 295 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

MacScience DIPLabo 32001 diffractometer
Absorption correction: none
6513 measured reflections
3597 independent reflections
3158 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.152
S = 1.14
3597 reflections
281 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O19—H19···O8^{i}	0.82	2.13	2.873 (3)	151
C2—H2B···O9	0.97	2.57	2.993 (4)	107
C3—H3B···O19	0.97	2.56	2.907 (3)	101
C6—H6A···O8	0.97	2.40	2.863 (4)	109
C12—H12···O8^{ii}	0.93	2.50	3.242 (3)	136
C15—H15···O9	0.93	2.46	2.845 (3)	105
C25—H25···O19	0.93	2.31	2.657 (4)	102
C31—H31···O19	0.93	2.40	2.764 (3)	103

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

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2179).

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supplementary materials

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[1-(4-Chloro-2-fluorophenylsulfonyl)piperidin-4-yl]diphenylmethanol

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Comment

The piperidine scaffold and its analogues are important pharmacophores that can be found in biologically active compounds across a number of different therapeutic areas; these include antiacetylcholinesterase (Sugimoto *et al.*, 1990), anti-HIV (Mao *et al.*, 1998), anticancer (Henderson *et al.*, 1996), antimicrobial, anti-implantation, anti-inflammatory and antioxidative activities. The benzhydrol piperidine is a basic component of the antihistamine drug Terefenadine which is used for the treatment of various allergic disorders (Li *et al.*, 2006). The piperidine sulfonamides are regarded as the most promising compounds in terms of both receptor affinity and subtype selectivity. Moreover, the M1 and M3 receptor affinity are quite sensitive to different substitutuents on the nitrogen of the piperidine ring. As a part of our ongoing research on novel bioactive heterocycles, the title compound was synthesized by the condensation of [piperidin-4-yl]-diphenylmethanol with 4-chloro-2-fluorobenzenesulfonyl chloride, with dichloromethane as solvent and triethylamine as the base. Here we report its crystal structure.

A perspective view of the title compound is shown in Fig. 1. A study of torsion angles, asymmetry parameters and least-squares plane calculations reveal that the piperidine ring is in a chair conformation. This has been confirmed by the puckering paramaters q_2 = 0.0382 (27) Å, q_3 = −0.5732 (26) Å, Q_T = 0.5746 (26) Å, θ = 175.98 (27)° and φ = 109 (4)° (Cremer & Pople, 1975). The conformation of the attachment of the diphenylmethyl and the sulfonyl groups to the piperidine ring are best described by the torsion angle values of −141.45 (19)° and 179.39 (19)° for S7—N1—C2—C3 and C18—C4—C5—C6, respectively; *i.e.* they adopt -antiperiplanar and +antiperiplanar conformations, respectively. The sulfonyl and diphenylmethanol groups are in equatorial positions.

The bond angles about the S atom shows significant deviation from that of a regular tetrahedron, with the largest deviations being observed for O8—S7—O9 [119.4 (1)°] and 09—S7—C10 [105.1 (1)°]. The widening of O8—S7—O9 is due to the repulsive interactions between the S═O bonds and the non-bonded interactions involving the two S═O bonds and the varied steric hindrance of the substituents. The structure thus has less steric interference. The bond angle N1—S7—C10 is comparable with the classic tetrahedral value of 109.47°. The sulfonyl O atoms, O8 and O9, are oriented in -synperiplanar and +synclinal conformations, respectively, as indicated by the torsion angle values of −21.5 (2)° and 49.9 (2)° for C6—N1—S7—O8 and C2—N1—S7—O9, respectively.

The dihedral angle between the least-squares plane, P1, defined by the atoms C2/C3/C5/C6 of the piperidine ring, and the benzene ring (C10–C15) is 49.80 (1)°. The dihedral angles between P1 and the phenyl rings (C20–C25) and (C26–C31) are 59.34 (1)° and 73.81 (1)°, respectively. The two phenyl rings make a dihedral angle of 65.13 (14)°.

The structure exhibits intermolecular hydrogen bonds of the type O—H···O and C—H···O. The molecules exhibit layered stacking and they form a one-dimensional polymeric chain (Figure 2).

Experimental

[Piperidin-4-yl]-diphenylmethanol (1.0 g, 3.74 mmol). was dissolved in dichloromethane (10 ml) and cooled to 0–5°C in an ice bath. Triethylamine (1.136 g, 11.22 mmol) was then added to the cold reaction mixture and stirred for 10 minutes. 4-Chloro-2-fluorobenzenesulfonyl chloride (0.852 g, 3.74 mmol) was then added to the reaction mixture. The reaction mixture was stirred at room temperature for 5 h. The reaction mass was monitored by TLC. After completion of the reaction, the solvent was removed under reduced pressure and the residue was taken up in water and extracted with ethyl acetate. The organic layer was washed with water and dried over anhydrous sodium sulfate. A white crystalline solid was obtained which was kept in ethyl acetate: methanol (3:1) for three days. Pure light yellow crystals were obtained by slow evaporation of the solvent.

Refinement

H atoms were placed at idealized positions and allowed to ride on their parent atoms with C—H distances in the range 0.92–0.97 Å and O—H = 0.82 Å; U_{iso}(H) values were set equal to 1.2U_{eq}(carrier atom).

Figures

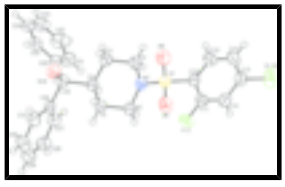

Fig. 1. The molecular structure, with atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

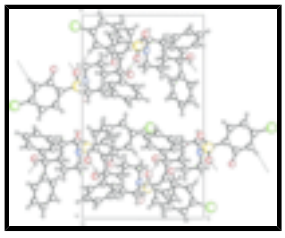

Fig. 2. The crystal packing in the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

[1-(4-Chloro-2-fluorophenylsulfonyl)piperidin-4-yl]diphenylmethanol

Crystal data

$C_{24}H_{23}ClFNO_3S$	F_{000} = 960
M_r = 459.94	D_x = 1.389 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6513 reflections
a = 10.238 (7) Å	θ = 2.1–25.0º
b = 11.295 (4) Å	μ = 0.30 mm^{-1}
c = 21.072 (12) Å	T = 295 (2) K
β = 115.481 (2)º	Block, pale yellow
V = 2200 (2) Å^3	0.25 × 0.20 × 0.20 mm

$Z = 4$

Data collection

MacScience DIPLabo 32001 diffractometer	3158 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.017$
Monochromator: graphite	$\theta_{max} = 25.0°$
$T = 295(2)$ K	$\theta_{min} = 2.1°$
ω scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = -12 \rightarrow 12$
6513 measured reflections	$l = -25 \rightarrow 24$
3597 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 0.6199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.152$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.14$	$\Delta\rho_{max} = 0.56$ e Å^{-3}
3597 reflections	$\Delta\rho_{min} = -0.43$ e Å^{-3}
281 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.037 (3)
Secondary atom site location: difference Fourier map	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All e.s.d.'s are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for All reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of F^2 > 2sigma(F^2) is used only for calculating –*R*-factor-obs *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}
Cl16	−0.08080 (10)	1.05994 (7)	0.05424 (5)	0.0803 (4)
S7	−0.08950 (6)	0.53792 (5)	0.15198 (3)	0.0418 (2)
F17	0.10550 (18)	0.73512 (14)	0.23283 (7)	0.0712 (6)
O8	−0.1120 (2)	0.54723 (16)	0.21419 (9)	0.0548 (6)

O9	−0.19477 (19)	0.47903 (16)	0.09182 (10)	0.0603 (6)
O19	0.27293 (18)	0.10628 (14)	0.20719 (9)	0.0479 (6)
N1	0.0630 (2)	0.47425 (17)	0.17395 (9)	0.0438 (6)
C2	0.1166 (3)	0.4518 (2)	0.12076 (12)	0.0489 (8)
C3	0.1912 (3)	0.3319 (2)	0.13428 (12)	0.0464 (8)
C4	0.3085 (2)	0.31985 (18)	0.20955 (11)	0.0361 (7)
C5	0.2446 (2)	0.3494 (2)	0.26104 (11)	0.0401 (7)
C6	0.1768 (3)	0.4716 (2)	0.24667 (11)	0.0430 (7)
C10	−0.0811 (2)	0.68354 (19)	0.12337 (11)	0.0388 (7)
C11	0.0136 (2)	0.7674 (2)	0.16736 (11)	0.0434 (7)
C12	0.0173 (3)	0.8821 (2)	0.14723 (13)	0.0495 (8)
C13	−0.0794 (3)	0.9146 (2)	0.08032 (13)	0.0509 (8)
C14	−0.1736 (3)	0.8345 (2)	0.03464 (14)	0.0641 (10)
C15	−0.1749 (3)	0.7192 (2)	0.05628 (13)	0.0568 (8)
C18	0.3811 (2)	0.19532 (18)	0.22271 (11)	0.0376 (7)
C20	0.4514 (2)	0.17185 (19)	0.17263 (11)	0.0400 (7)
C21	0.5273 (3)	0.2577 (2)	0.15578 (14)	0.0551 (9)
C22	0.5916 (3)	0.2331 (3)	0.11130 (15)	0.0708 (11)
C23	0.5801 (3)	0.1220 (3)	0.08268 (15)	0.0724 (13)
C24	0.5047 (4)	0.0364 (3)	0.09887 (14)	0.0672 (11)
C25	0.4409 (3)	0.0603 (2)	0.14300 (12)	0.0506 (8)
C26	0.4948 (2)	0.18438 (19)	0.29986 (11)	0.0383 (7)
C27	0.6161 (3)	0.2559 (2)	0.32666 (12)	0.0464 (8)
C28	0.7121 (3)	0.2531 (2)	0.39708 (14)	0.0565 (9)
C29	0.6895 (3)	0.1771 (3)	0.44252 (14)	0.0596 (9)
C30	0.5722 (3)	0.1037 (3)	0.41678 (15)	0.0648 (10)
C31	0.4752 (3)	0.1064 (2)	0.34611 (14)	0.0531 (9)
H2A	0.18440	0.51330	0.12280	0.0590*
H2B	0.03660	0.45280	0.07430	0.0590*
H3A	0.23440	0.32080	0.10180	0.0560*
H3B	0.11960	0.27010	0.12540	0.0560*
H4	0.38350	0.37880	0.21600	0.0430*
H5A	0.17180	0.29100	0.25680	0.0480*
H5B	0.32020	0.34620	0.30870	0.0480*
H6A	0.13540	0.48870	0.27930	0.0520*
H6B	0.24970	0.53090	0.25270	0.0520*
H12	0.08280	0.93630	0.17760	0.0590*
H14	−0.23640	0.85760	−0.01070	0.0770*
H15	−0.23940	0.66500	0.02540	0.0680*
H19	0.23170	0.11500	0.23270	0.0720*
H21	0.53540	0.33320	0.17460	0.0660*
H22	0.64270	0.29200	0.10080	0.0850*
H23	0.62300	0.10520	0.05270	0.0870*
H24	0.49660	−0.03890	0.07970	0.0800*
H25	0.38990	0.00090	0.15320	0.0610*
H27	0.63370	0.30720	0.29660	0.0560*
H28	0.79230	0.30280	0.41380	0.0680*
H29	0.75320	0.17590	0.49000	0.0720*
H30	0.55710	0.05110	0.44690	0.0780*

H31	0.39620	0.05550	0.32960	0.0640*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl16	0.0941 (7)	0.0507 (5)	0.1008 (7)	0.0099 (4)	0.0463 (5)	0.0212 (4)
S7	0.0346 (4)	0.0436 (4)	0.0460 (4)	0.0033 (2)	0.0161 (3)	0.0028 (2)
F17	0.0733 (11)	0.0630 (10)	0.0444 (8)	−0.0008 (8)	−0.0059 (7)	−0.0002 (7)
O8	0.0504 (11)	0.0641 (11)	0.0610 (11)	0.0160 (8)	0.0345 (9)	0.0157 (8)
O9	0.0451 (10)	0.0527 (11)	0.0667 (12)	−0.0078 (8)	0.0084 (9)	−0.0041 (8)
O19	0.0460 (10)	0.0397 (9)	0.0607 (10)	−0.0104 (7)	0.0255 (8)	−0.0049 (7)
N1	0.0410 (11)	0.0519 (11)	0.0390 (10)	0.0128 (8)	0.0178 (8)	0.0027 (8)
C2	0.0546 (15)	0.0546 (14)	0.0417 (12)	0.0149 (11)	0.0246 (11)	0.0083 (10)
C3	0.0519 (15)	0.0503 (13)	0.0394 (12)	0.0105 (10)	0.0220 (10)	−0.0002 (9)
C4	0.0339 (12)	0.0356 (11)	0.0414 (11)	0.0018 (8)	0.0188 (9)	0.0001 (8)
C5	0.0386 (12)	0.0452 (12)	0.0381 (11)	0.0057 (9)	0.0181 (9)	0.0021 (9)
C6	0.0401 (13)	0.0468 (13)	0.0406 (12)	0.0076 (9)	0.0158 (10)	−0.0017 (9)
C10	0.0351 (12)	0.0424 (12)	0.0383 (11)	0.0044 (9)	0.0152 (9)	−0.0014 (9)
C11	0.0395 (13)	0.0500 (13)	0.0354 (11)	0.0060 (10)	0.0110 (9)	−0.0019 (9)
C12	0.0490 (15)	0.0433 (13)	0.0549 (14)	0.0004 (10)	0.0212 (11)	−0.0081 (10)
C13	0.0549 (16)	0.0459 (13)	0.0564 (14)	0.0093 (11)	0.0282 (12)	0.0063 (11)
C14	0.0705 (19)	0.0584 (17)	0.0466 (14)	0.0072 (13)	0.0094 (13)	0.0135 (12)
C15	0.0583 (16)	0.0513 (14)	0.0421 (13)	0.0004 (12)	0.0040 (11)	0.0007 (10)
C18	0.0350 (12)	0.0326 (11)	0.0474 (12)	−0.0032 (8)	0.0198 (10)	−0.0009 (8)
C20	0.0362 (12)	0.0434 (12)	0.0396 (11)	0.0064 (9)	0.0155 (9)	−0.0006 (9)
C21	0.0556 (16)	0.0598 (16)	0.0615 (16)	−0.0057 (12)	0.0361 (13)	−0.0075 (12)
C22	0.0570 (18)	0.105 (2)	0.0639 (17)	−0.0040 (16)	0.0389 (15)	−0.0023 (16)
C23	0.0595 (19)	0.113 (3)	0.0496 (15)	0.0229 (18)	0.0282 (14)	−0.0076 (16)
C24	0.073 (2)	0.076 (2)	0.0435 (14)	0.0273 (16)	0.0164 (13)	−0.0116 (12)
C25	0.0562 (15)	0.0463 (13)	0.0414 (12)	0.0124 (11)	0.0136 (11)	−0.0015 (9)
C26	0.0393 (13)	0.0343 (11)	0.0457 (12)	0.0060 (8)	0.0224 (10)	0.0015 (8)
C27	0.0477 (14)	0.0468 (13)	0.0477 (13)	−0.0047 (10)	0.0235 (11)	−0.0012 (10)
C28	0.0514 (15)	0.0620 (16)	0.0534 (15)	−0.0054 (12)	0.0201 (12)	−0.0068 (12)
C29	0.0582 (17)	0.0714 (18)	0.0469 (14)	0.0126 (13)	0.0204 (12)	0.0056 (12)
C30	0.0659 (19)	0.0725 (19)	0.0601 (16)	0.0094 (15)	0.0310 (14)	0.0219 (14)
C31	0.0479 (15)	0.0530 (15)	0.0604 (15)	0.0010 (11)	0.0252 (12)	0.0129 (11)

Geometric parameters (Å, °)

Cl16—C13	1.729 (2)	C26—C31	1.391 (3)
S7—O8	1.429 (2)	C26—C27	1.382 (4)
S7—O9	1.427 (2)	C27—C28	1.383 (4)
S7—N1	1.596 (2)	C28—C29	1.378 (4)
S7—C10	1.767 (2)	C29—C30	1.365 (5)
F17—C11	1.343 (3)	C30—C31	1.390 (4)
O19—C18	1.426 (3)	C2—H2A	0.9698
O19—H19	0.8198	C2—H2B	0.9693
N1—C2	1.467 (3)	C3—H3A	0.9696
N1—C6	1.473 (3)	C3—H3B	0.9704

C2—C3	1.520 (3)	C4—H4	0.9806
C3—C4	1.529 (3)	C5—H5A	0.9702
C4—C5	1.526 (3)	C5—H5B	0.9696
C4—C18	1.559 (3)	C6—H6A	0.9698
C5—C6	1.516 (3)	C6—H6B	0.9696
C10—C11	1.387 (3)	C12—H12	0.9299
C10—C15	1.384 (3)	C14—H14	0.9304
C11—C12	1.369 (3)	C15—H15	0.9298
C12—C13	1.379 (4)	C21—H21	0.9290
C13—C14	1.370 (4)	C22—H22	0.9300
C14—C15	1.382 (3)	C23—H23	0.9310
C18—C26	1.544 (3)	C24—H24	0.9298
C18—C20	1.535 (3)	C25—H25	0.9303
C20—C21	1.381 (4)	C27—H27	0.9315
C20—C25	1.390 (3)	C28—H28	0.9298
C21—C22	1.386 (4)	C29—H29	0.9301
C22—C23	1.375 (5)	C30—H30	0.9296
C23—C24	1.369 (5)	C31—H31	0.9293
C24—C25	1.374 (5)		
Cl16···Cl16^{i}	3.6125 (15)	C31···H5B	3.0659
Cl16···H3B^{ii}	3.0734	C31···H19	2.6094
Cl16···H23^{iii}	3.0617	H2A···H6B	2.5307
Cl16···H28^{iv}	3.0889	H2A···C28^{x}	3.0027
F17···O8	2.977 (3)	H2A···C29^{x}	2.9177
F17···N1	3.154 (2)	H2B···O9	2.5666
F17···C6	3.049 (3)	H3A···C20	2.6722
F17···H6B	2.6728	H3A···C21	2.8034
O8···F17	2.977 (3)	H3B···Cl16^{ix}	3.0734
O8···O19^{v}	2.873 (3)	H3B···O19	2.5555
O8···C12^{vi}	3.242 (3)	H3B···H5A	2.5951
O9···C29^{vii}	3.347 (3)	H4···C21	2.6937
O19···O8^{vi}	2.873 (3)	H4···C27	2.8736
O8···H6A	2.3968	H4···H6B	2.5174
O8···H31^{v}	2.6488	H4···H21	2.1476
O8···H12^{vi}	2.5044	H4···H27	2.5213
O8···H19^{v}	2.1261	H5A···O19	2.7293
O9···H22^{viii}	2.7456	H5A···H3B	2.5951
O9···H2B	2.5666	H5A···H19	2.2031
O9···H15	2.4555	H5A···C11^{vi}	2.9729
O9···H29^{vii}	2.6393	H5B···C26	2.6186
O19···H3B	2.5555	H5B···C27	3.0618
O19···H5A	2.7293	H5B···C31	3.0659
O19···H25	2.3059	H5B···C24^{x}	2.9357
O19···H31	2.4030	H6A···O8	2.3968
O19···H12^{ix}	2.6114	H6A···C12^{vi}	2.8936

N1···F17	3.154 (2)	H6B···F17	2.6728
C3···C21	3.377 (5)	H6B···H2A	2.5307
C5···C31	3.561 (3)	H6B···H4	2.5174
C6···F17	3.049 (3)	H6B···C24^{x}	3.0565
C6···C24^{x}	3.554 (4)	H6B···C25^{x}	2.9974
C12···O8^{v}	3.242 (3)	H12···O19^{ii}	2.6114
C12···C28^{x}	3.591 (4)	H12···H19^{ii}	2.4946
C21···C3	3.377 (5)	H12···O8^{v}	2.5044
C21···C27	3.313 (4)	H14···C24^{xiii}	2.8354
C24···C6^{xi}	3.554 (4)	H14···C25^{xiii}	2.8272
C25···C27^{xi}	3.590 (3)	H15···O9	2.4555
C27···C21	3.313 (4)	H19···C5	2.7048
C27···C25^{x}	3.590 (3)	H19···C31	2.6094
C28···C12^{xi}	3.591 (4)	H19···H5A	2.2031
C29···O9^{xii}	3.347 (3)	H19···H12^{ix}	2.4946
C31···C5	3.561 (3)	H19···H31	2.1210
C4···H27	3.0357	H19···O8^{vi}	2.1261
C4···H21	2.7264	H21···C4	2.7264
C5···H19	2.7048	H21···C27	3.0715
C11···H5A^{v}	2.9729	H21···H4	2.1476
C12···H28^{x}	2.9005	H21···H27	2.3413
C12···H6A^{v}	2.8936	H22···O9^{xiv}	2.7456
C20···H27	2.9068	H23···Cl16^{xv}	3.0617
C20···H3A	2.6722	H25···O19	2.3059
C21···H4	2.6937	H25···C27^{xi}	2.8043
C21···H3A	2.8034	H25···C28^{xi}	3.0158
C21···H27	2.7428	H25···H27^{xi}	2.4867
C24···H6B^{xi}	3.0565	H27···C4	3.0357
C24···H5B^{xi}	2.9357	H27···C20	2.9068
C24···H14^{xiii}	2.8354	H27···C21	2.7428
C25···H14^{xiii}	2.8272	H27···H4	2.5213
C25···H6B^{xi}	2.9974	H27···H21	2.3413
C26···H5B	2.6186	H27···H25^{x}	2.4867
C27···H4	2.8736	H28···C12^{xi}	2.9005
C27···H5B	3.0618	H28···Cl16^{xvi}	3.0889
C27···H21	3.0715	H29···O9^{xii}	2.6393
C27···H25^{x}	2.8043	H31···O19	2.4030
C28···H2A^{xi}	3.0027	H31···H19	2.1210
C28···H25^{x}	3.0158	H31···O8^{vi}	2.6488
C29···H2A^{xi}	2.9177		
O8—S7—O9	119.41 (12)	C26—C31—C30	120.8 (3)
O8—S7—N1	106.90 (11)	N1—C2—H2A	109.77

O8—S7—C10	107.17 (11)	N1—C2—H2B	109.78
O9—S7—N1	108.57 (11)	C3—C2—H2A	109.72
O9—S7—C10	105.07 (11)	C3—C2—H2B	109.74
N1—S7—C10	109.47 (11)	H2A—C2—H2B	108.31
C18—O19—H19	109.45	C2—C3—H3A	109.14
S7—N1—C6	123.24 (17)	C2—C3—H3B	109.13
C2—N1—C6	113.8 (2)	C4—C3—H3A	109.12
S7—N1—C2	119.82 (15)	C4—C3—H3B	109.09
N1—C2—C3	109.50 (19)	H3A—C3—H3B	107.88
C2—C3—C4	112.38 (18)	C3—C4—H4	107.58
C3—C4—C5	109.4 (2)	C5—C4—H4	107.62
C5—C4—C18	112.85 (17)	C18—C4—H4	107.64
C3—C4—C18	111.50 (17)	C4—C5—H5A	109.47
C4—C5—C6	110.90 (18)	C4—C5—H5B	109.45
N1—C6—C5	108.39 (18)	C6—C5—H5A	109.45
S7—C10—C15	120.36 (17)	C6—C5—H5B	109.50
C11—C10—C15	117.5 (2)	H5A—C5—H5B	108.02
S7—C10—C11	122.04 (16)	N1—C6—H6A	110.01
F17—C11—C12	118.3 (2)	N1—C6—H6B	110.00
C10—C11—C12	123.0 (2)	C5—C6—H6A	110.00
F17—C11—C10	118.7 (2)	C5—C6—H6B	110.02
C11—C12—C13	117.7 (2)	H6A—C6—H6B	108.43
Cl16—C13—C12	118.80 (19)	C11—C12—H12	121.18
Cl16—C13—C14	119.8 (2)	C13—C12—H12	121.09
C12—C13—C14	121.4 (2)	C13—C14—H14	120.19
C13—C14—C15	119.6 (2)	C15—C14—H14	120.18
C10—C15—C14	120.7 (2)	C10—C15—H15	119.68
O19—C18—C20	105.56 (17)	C14—C15—H15	119.63
O19—C18—C26	110.14 (17)	C20—C21—H21	119.44
C4—C18—C20	111.24 (17)	C22—C21—H21	119.45
C4—C18—C26	110.19 (17)	C21—C22—H22	119.81
C20—C18—C26	110.24 (18)	C23—C22—H22	119.95
O19—C18—C4	109.37 (18)	C22—C23—H23	120.48
C18—C20—C21	122.5 (2)	C24—C23—H23	120.43
C21—C20—C25	117.7 (2)	C23—C24—H24	119.49
C18—C20—C25	119.8 (2)	C25—C24—H24	119.63
C20—C21—C22	121.1 (2)	C20—C25—H25	119.52
C21—C22—C23	120.2 (3)	C24—C25—H25	119.49
C22—C23—C24	119.1 (3)	C26—C27—H27	119.24
C23—C24—C25	120.9 (3)	C28—C27—H27	119.18
C20—C25—C24	121.0 (3)	C27—C28—H28	119.78
C18—C26—C27	121.41 (19)	C29—C28—H28	119.86
C27—C26—C31	117.3 (2)	C28—C29—H29	120.47
C18—C26—C31	121.2 (2)	C30—C29—H29	120.56
C26—C27—C28	121.6 (2)	C29—C30—H30	119.55
C27—C28—C29	120.4 (3)	C31—C30—H30	119.57
C28—C29—C30	119.0 (3)	C26—C31—H31	119.62
C29—C30—C31	120.9 (3)	C30—C31—H31	119.54
O8—S7—N1—C2	179.94 (17)	C10—C11—C12—C13	0.8 (4)

O9—S7—N1—C2	49.9 (2)	C11—C12—C13—C14	−1.7 (5)
C10—S7—N1—C2	−64.3 (2)	C11—C12—C13—Cl16	178.2 (2)
O8—S7—N1—C6	−21.5 (2)	Cl16—C13—C14—C15	−178.3 (2)
O9—S7—N1—C6	−151.58 (19)	C12—C13—C14—C15	1.7 (5)
C10—S7—N1—C6	94.2 (2)	C13—C14—C15—C10	−0.6 (5)
N1—S7—C10—C11	−61.7 (2)	C4—C18—C26—C31	113.2 (2)
O8—S7—C10—C11	53.8 (2)	C26—C18—C20—C25	97.5 (2)
O9—S7—C10—C11	−178.2 (2)	O19—C18—C26—C27	175.9 (2)
N1—S7—C10—C15	121.2 (2)	O19—C18—C26—C31	−7.6 (3)
O8—S7—C10—C15	−123.2 (2)	C4—C18—C26—C27	−63.4 (3)
O9—S7—C10—C15	4.8 (2)	O19—C18—C20—C21	159.2 (2)
S7—N1—C2—C3	−141.45 (19)	C20—C18—C26—C27	59.8 (3)
S7—N1—C6—C5	139.33 (19)	C20—C18—C26—C31	−123.7 (2)
C2—N1—C6—C5	−61.0 (3)	C4—C18—C20—C21	40.7 (3)
C6—N1—C2—C3	58.1 (3)	C26—C18—C20—C21	−81.9 (3)
N1—C2—C3—C4	−53.4 (3)	C4—C18—C20—C25	−140.0 (2)
C2—C3—C4—C5	53.2 (3)	O19—C18—C20—C25	−21.4 (3)
C2—C3—C4—C18	178.7 (2)	C21—C20—C25—C24	0.4 (4)
C3—C4—C18—C20	59.6 (2)	C25—C20—C21—C22	−0.5 (4)
C3—C4—C18—C26	−177.9 (2)	C18—C20—C25—C24	−179.0 (2)
C5—C4—C18—O19	67.0 (2)	C18—C20—C21—C22	178.9 (2)
C5—C4—C18—C20	−176.80 (18)	C20—C21—C22—C23	0.3 (4)
C5—C4—C18—C26	−54.2 (2)	C21—C22—C23—C24	−0.1 (5)
C18—C4—C5—C6	179.39 (19)	C22—C23—C24—C25	0.1 (5)
C3—C4—C18—O19	−56.6 (2)	C23—C24—C25—C20	−0.2 (5)
C3—C4—C5—C6	−55.8 (2)	C27—C26—C31—C30	1.8 (4)
C4—C5—C6—N1	58.8 (3)	C18—C26—C27—C28	174.7 (2)
S7—C10—C15—C14	176.9 (2)	C18—C26—C31—C30	−174.9 (3)
C11—C10—C15—C14	−0.3 (4)	C31—C26—C27—C28	−2.0 (4)
C15—C10—C11—C12	0.2 (4)	C26—C27—C28—C29	0.7 (4)
C15—C10—C11—F17	179.8 (2)	C27—C28—C29—C30	0.9 (5)
S7—C10—C11—C12	−177.0 (2)	C28—C29—C30—C31	−1.1 (5)
S7—C10—C11—F17	2.7 (3)	C29—C30—C31—C26	−0.3 (5)
F17—C11—C12—C13	−178.8 (3)		

Symmetry codes: (i) −*x*, −*y*+2, −*z*; (ii) *x*, *y*+1, *z*; (iii) *x*−1, *y*+1, *z*; (iv) *x*−1, −*y*+3/2, *z*−1/2; (v) −*x*, *y*+1/2, −*z*+1/2; (vi) −*x*, *y*−1/2, −*z*+1/2; (vii) *x*−1, −*y*+1/2, *z*−1/2; (viii) *x*−1, *y*, *z*; (ix) *x*, *y*−1, *z*; (x) −*x*+1, *y*+1/2, −*z*+1/2; (xi) −*x*+1, *y*−1/2, −*z*+1/2; (xii) *x*+1, −*y*+1/2, *z*+1/2; (xiii) −*x*, −*y*+1, −*z*; (xiv) *x*+1, *y*, *z*; (xv) *x*+1, *y*−1, *z*; (xvi) *x*+1, −*y*+3/2, *z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O19—H19···O8^{vi}	0.82	2.13	2.873 (3)	151
C2—H2B···O9	0.97	2.57	2.993 (4)	107
C3—H3B···O19	0.97	2.56	2.907 (3)	101
C6—H6A···O8	0.97	2.40	2.863 (4)	109
C12—H12···O8^{v}	0.93	2.50	3.242 (3)	136
C15—H15···O9	0.93	2.46	2.845 (3)	105
C25—H25···O19	0.93	2.31	2.657 (4)	102
C31—H31···O19	0.93	2.40	2.764 (3)	103

Symmetry codes: (vi) −*x*, *y*−1/2, −*z*+1/2; (v) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

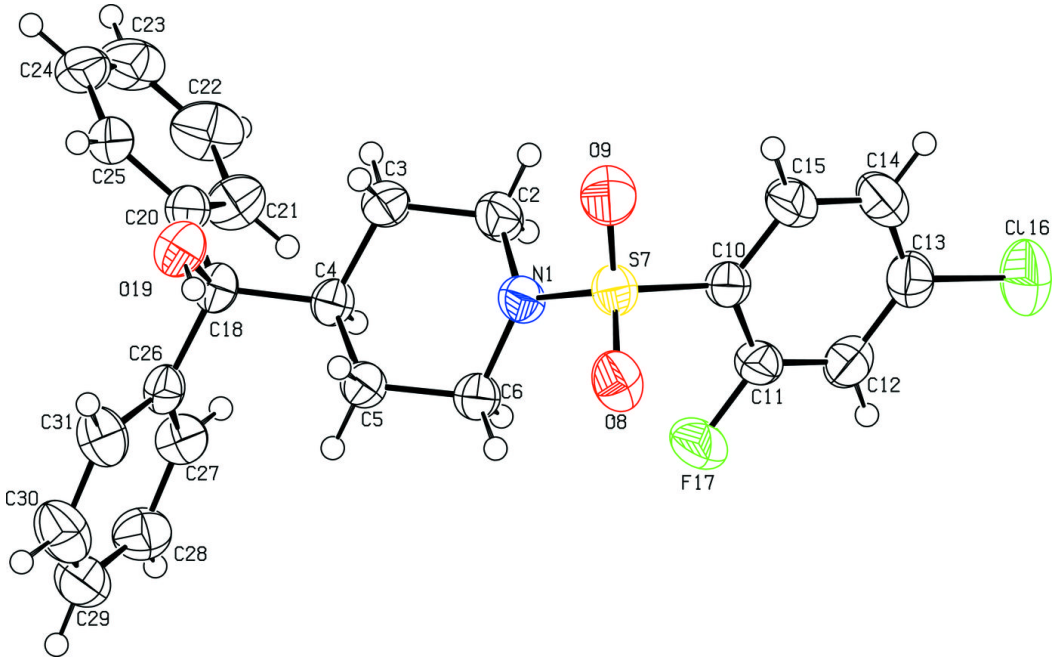

Fig. 2