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Crystal structure of 2-amino-*N*-(2-fluorophenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

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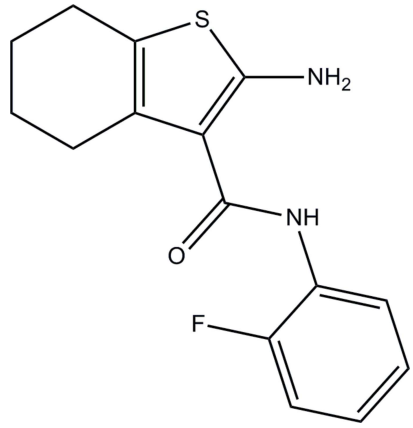
In the title compound, $C_{15}H_{15}FN_2OS$, the dihedral angle between the planes of the benzothiophene ring system and the fluorobenzene ring is 3.74 (14)°. The six-membered ring of the benzothiophene moiety adopts a half-chair conformation. The molecular conformation is consolidated by intramolecular N—H···F and N—H···O hydrogen bonds. In the crystal, molecules are linked by N—H···O hydrogen bonds, generating *C*(6) [001] chains.

Keywords: crystal structure; benzothiophene derivative; biological properties; hydrogen bonding.

CCDC reference: 1045467

1. Related literature

For background to thiophene derivatives, see: Bonini *et al.* (2005); Brault *et al.* (2005); Isloor *et al.* (2010). For intermolecular interactions involving F atoms, see: Choudhury *et al.* (2004).



2. Experimental

2.1. Crystal data

$C_{15}H_{15}FN_2OS$
M_r = 290.36
Monoclinic *Cc*
a = 11.213 (13) Å
b = 14.231 (17) Å
c = 9.582 (15) Å
β = 116.76 (3)°
V = 1365 (3) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.25 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

2.2. Data collection

Bruker APEXII CCD area-detector diffractometer
5264 measured reflections
2577 independent reflections
2363 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.081
S = 1.84
2577 reflections
182 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.06 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H9*A*···F7	0.86	2.26	2.643 (5)	107
N16—H15*C*···O10	0.86	2.16	2.733 (5)	124
N16—H15*D*···O10^{i}	0.86	2.25	2.986 (6)	143

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7493).

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supporting information

Acta Cryst. (2015). E**71**, o807–o808 [https://doi.org/10.1107/S2056989015018022]

Crystal structure of 2-amino-*N*-(2-fluorophenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

K. Chandra Kumar, V. Umesh, T. K. Madhura, B. M. Rajesh and Chandra

S1. Comment

Thiophene nucleus has been established as a potential entity in the largely growing chemical world of heterocyclic compounds possessing promising pharmacological characteristics such as anti-HIV PR inhibitors (Bonini *et al.*, 2005) and anti-breast cancer (Brault *et al.*, 2005) activities. Particularly, benzothiophene derivative shows significant antimicrobial and anti- inflammatory activities (Isloora *et al.*, 2010). In addition structures containing fluorine atoms plays a major role in intermolecular interactions (Choudhury *et al.*, 2004). The title compound was prepared and characterized by single-crystal X-ray diffraction studies.

In the molecular structure of the title compound (Fig. 1), the dihedral angle between the flurobenzene (C1–C2–C3–C4–C5–C6) and benzothiophene (C11–C12–C13–S14–C15–C17–C18–C19–C20) ring is 3.74 (14)°. The benzothiophene moiety adopts a half chair conformation conformation with puckering parameter Q = 0.475 (3) Å and φ = 215.4 (5)°, and the maximum deviation found on the puckered atom at C18 is 0.372 (4) Å. The carboximidamide unit is in anti-periplanar conformation with respect to the benzothiophene moiety, as indicated by the torsion angle value of 161.9 (3)° (N8–C9–C11–C15). The crystal structure features intermolecular N—H···O hydrogen bonds. The packing diagram of the molecule viewed down the *a* axis as shown in Fig. 2.

S2. Experimental

Cyclohexanone (1 equiv.), 2-cyano-*N*-(2-fluorophenyl) acetamide (1.1 equiv.), elemental sulfur (1.2 equiv.), diethylamine (0.8 equiv.) was taken in ethanol and mixed thoroughly in a microwave tube. The tube was sealed and irradiated at 325 K for 15 min. After cooling ethyl acetate was added to the reaction mixture and solid residue was removed by filtration. The filtrate was concentrated under reduced pressure and purified by column chromatography to obtain yellow block shaped crystals.

S3. Refinement

H atoms were placed at idealized positions and allowed to ride on their parent atoms with N–H distance is equal to 0.86 and C–H distances in the range of 0.93 to 0.97 Å; U_{iso}(H) = 1.2–1.5U_{eq}(carrier atom) for all H atoms.

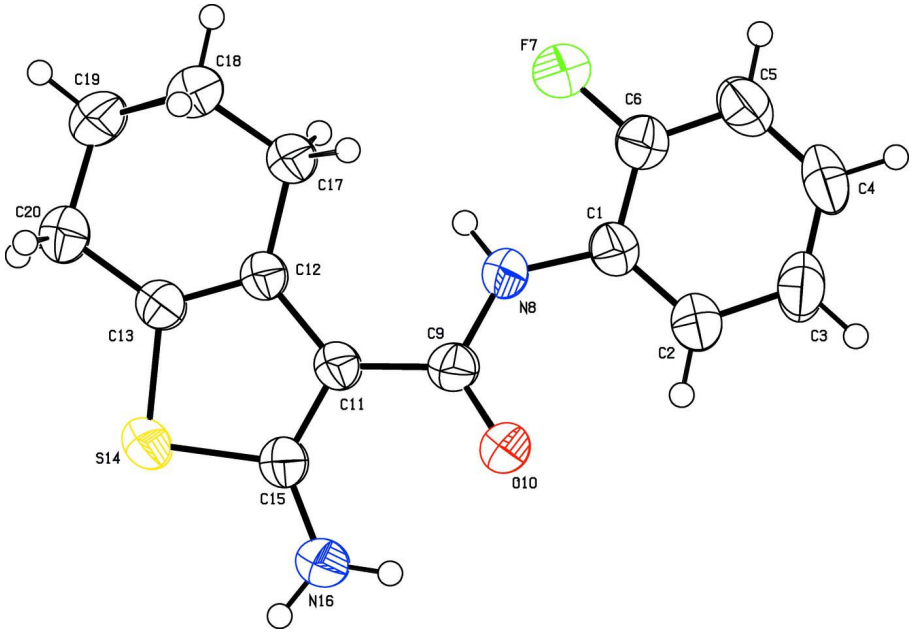

Figure 1

Perspective diagram of the molecule with 50% probability displacement ellipsoids.

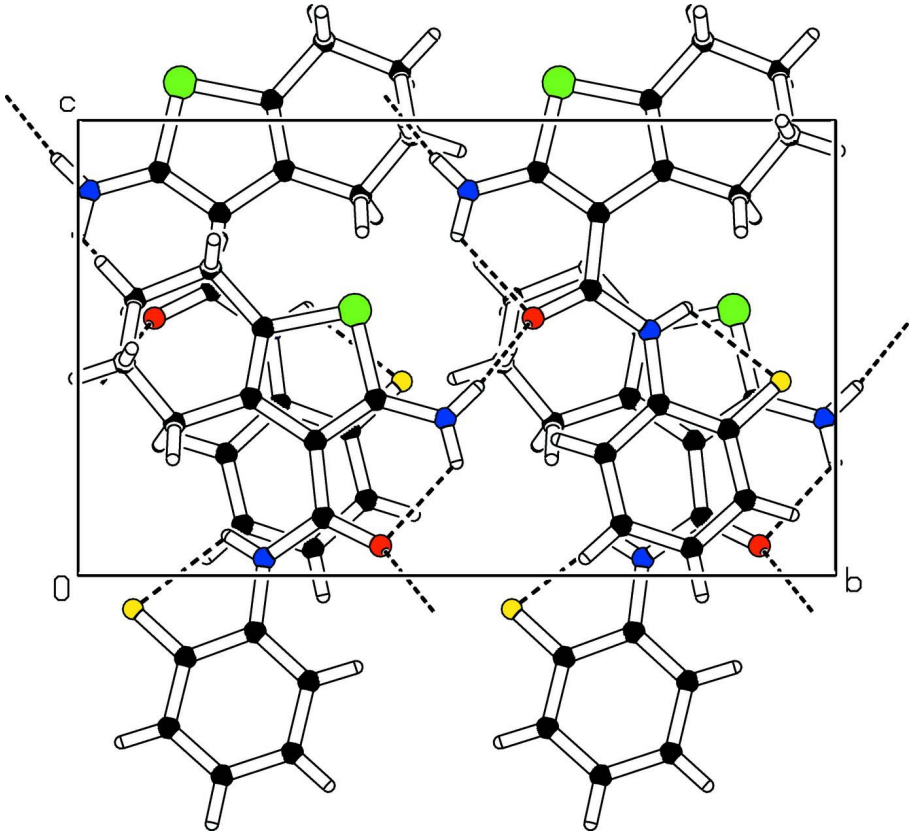

Figure 2

Packing diagram of the molecule viewed down the 'a′ axis.

2-Amino-*N*-(2-fluorophenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

Crystal data

$C_{15}H_{15}FN_2OS$
$M_r = 290.36$
Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 11.213$ (13) Å
$b = 14.231$ (17) Å
$c = 9.582$ (15) Å
$\beta = 116.76$ (3)°
$V = 1365$ (3) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2577 reflections
$\theta = 2.5$–26.4°
$\mu = 0.25$ mm^{-1}
$T = 293$ K
Bolck, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
ω and φ scans
5264 measured reflections
2577 independent reflections
2363 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.081$
$S = 1.84$
2577 reflections
182 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.010P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983), **??? Friedel pairs**
Absolute structure parameter: 0.06 (7)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su'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 $F^2 > \sigma(F^2)$ 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}
S14	0.42139 (7)	0.13494 (4)	1.08277 (7)	0.0586 (3)
F7	0.17969 (19)	0.42740 (10)	0.4264 (2)	0.0746 (6)
O10	0.2917 (2)	0.10081 (11)	0.56571 (19)	0.0568 (7)
N8	0.2299 (2)	0.25423 (14)	0.5376 (2)	0.0503 (7)

N16	0.3503 (2)	0.01559 (15)	0.8447 (3)	0.0674 (9)
C1	0.1812 (2)	0.26650 (18)	0.3762 (3)	0.0466 (9)
C2	0.1530 (3)	0.1953 (2)	0.2671 (3)	0.0588 (10)
C3	0.1087 (4)	0.2175 (3)	0.1106 (3)	0.0727 (11)
C4	0.0865 (3)	0.3097 (3)	0.0601 (4)	0.0735 (13)
C5	0.1109 (3)	0.3807 (2)	0.1661 (3)	0.0643 (11)
C6	0.1566 (3)	0.35800 (18)	0.3201 (3)	0.0519 (9)
C9	0.2880 (3)	0.17625 (17)	0.6279 (3)	0.0444 (9)
C11	0.3397 (2)	0.18699 (17)	0.7955 (3)	0.0423 (8)
C12	0.3705 (3)	0.27223 (17)	0.8901 (3)	0.0422 (8)
C13	0.4142 (3)	0.25543 (17)	1.0436 (3)	0.0478 (8)
C15	0.3637 (3)	0.10698 (17)	0.8869 (3)	0.0483 (9)
C17	0.3638 (3)	0.37266 (16)	0.8342 (3)	0.0497 (9)
C18	0.4470 (3)	0.43925 (17)	0.9684 (3)	0.0575 (10)
C19	0.4211 (4)	0.42420 (18)	1.1078 (3)	0.0665 (11)
C20	0.4583 (3)	0.32519 (19)	1.1743 (3)	0.0583 (10)
H2A	0.16370	0.13280	0.29880	0.0710*
H3A	0.09370	0.16960	0.03860	0.0870*
H4A	0.05520	0.32350	−0.04550	0.0880*
H5A	0.09670	0.44300	0.13370	0.0770*
H9A	0.22220	0.30220	0.58740	0.0600*
H15C	0.32330	0.00050	0.74820	0.0810*
H15D	0.36900	−0.02760	0.91420	0.0810*
H18A	0.55430	0.32090	1.23760	0.0700*
H18B	0.41570	0.31140	1.24040	0.0700*
H20A	0.32720	0.43510	1.07740	0.0800*
H20B	0.47240	0.46950	1.18820	0.0800*
H21A	0.54110	0.42920	0.99880	0.0690*
H21B	0.42570	0.50370	0.93290	0.0690*
H22A	0.27150	0.39340	0.78560	0.0600*
H22B	0.39610	0.37500	0.75590	0.0600*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S14	0.0914 (6)	0.0458 (4)	0.0421 (4)	0.0089 (4)	0.0332 (4)	0.0085 (3)
F7	0.1052 (14)	0.0470 (9)	0.0552 (10)	−0.0004 (8)	0.0217 (9)	−0.0020 (7)
O10	0.0853 (14)	0.0416 (10)	0.0432 (10)	0.0028 (9)	0.0286 (10)	−0.0026 (8)
N8	0.0726 (15)	0.0413 (11)	0.0362 (11)	0.0066 (10)	0.0239 (11)	−0.0015 (8)
N16	0.116 (2)	0.0397 (13)	0.0496 (13)	0.0007 (12)	0.0400 (13)	0.0038 (10)
C1	0.0467 (15)	0.0481 (16)	0.0397 (15)	0.0014 (11)	0.0147 (13)	0.0030 (11)
C2	0.073 (2)	0.0556 (17)	0.0419 (15)	0.0064 (14)	0.0206 (15)	0.0020 (12)
C3	0.087 (2)	0.080 (2)	0.0370 (15)	0.0112 (17)	0.0154 (15)	−0.0070 (15)
C4	0.095 (3)	0.083 (2)	0.0342 (15)	0.0095 (19)	0.0217 (16)	0.0113 (14)
C5	0.070 (2)	0.0614 (18)	0.0486 (18)	0.0004 (14)	0.0153 (15)	0.0137 (13)
C6	0.0546 (17)	0.0487 (16)	0.0459 (16)	−0.0034 (12)	0.0170 (13)	−0.0001 (12)
C9	0.0539 (17)	0.0385 (13)	0.0445 (14)	−0.0016 (12)	0.0255 (13)	0.0000 (11)
C11	0.0541 (17)	0.0391 (13)	0.0377 (13)	0.0027 (11)	0.0241 (13)	0.0029 (10)

C12	0.0534 (16)	0.0389 (13)	0.0379 (15)	0.0012 (11)	0.0238 (13)	0.0015 (10)
C13	0.0616 (16)	0.0414 (13)	0.0426 (15)	0.0068 (12)	0.0255 (14)	0.0037 (10)
C15	0.0671 (19)	0.0425 (14)	0.0408 (15)	0.0012 (12)	0.0293 (14)	0.0005 (11)
C17	0.0697 (17)	0.0404 (14)	0.0431 (13)	0.0014 (12)	0.0291 (12)	0.0014 (10)
C18	0.075 (2)	0.0433 (14)	0.0542 (16)	−0.0033 (13)	0.0292 (15)	−0.0045 (12)
C19	0.100 (2)	0.0474 (15)	0.059 (2)	0.0027 (16)	0.0418 (19)	−0.0082 (14)
C20	0.079 (2)	0.0536 (16)	0.0434 (16)	0.0055 (14)	0.0286 (15)	−0.0018 (12)

Geometric parameters (Å, º)

S14—C13	1.749 (4)	C12—C17	1.516 (4)
S14—C15	1.734 (4)	C12—C13	1.346 (4)
F7—C6	1.357 (4)	C13—C20	1.497 (4)
O10—C9	1.238 (4)	C17—C18	1.530 (4)
N8—C1	1.400 (4)	C18—C19	1.505 (5)
N8—C9	1.377 (4)	C19—C20	1.525 (4)
N16—C15	1.350 (4)	C2—H2A	0.9300
N8—H9A	0.8600	C3—H3A	0.9300
N16—H15C	0.8600	C4—H4A	0.9300
N16—H15D	0.8600	C5—H5A	0.9300
C1—C6	1.388 (4)	C17—H22A	0.9700
C1—C2	1.386 (4)	C17—H22B	0.9700
C2—C3	1.387 (4)	C18—H21A	0.9700
C3—C4	1.382 (6)	C18—H21B	0.9700
C4—C5	1.370 (5)	C19—H20A	0.9700
C5—C6	1.365 (4)	C19—H20B	0.9700
C9—C11	1.449 (4)	C20—H18A	0.9700
C11—C12	1.460 (4)	C20—H18B	0.9700
C11—C15	1.387 (4)		
C13—S14—C15	91.95 (12)	C12—C17—C18	111.9 (2)
C1—N8—C9	129.3 (2)	C17—C18—C19	111.7 (3)
C9—N8—H9A	115.00	C18—C19—C20	112.1 (3)
C1—N8—H9A	115.00	C13—C20—C19	109.8 (2)
H15C—N16—H15D	120.00	C1—C2—H2A	120.00
C15—N16—H15D	120.00	C3—C2—H2A	120.00
C15—N16—H15C	120.00	C2—C3—H3A	119.00
C2—C1—C6	117.1 (2)	C4—C3—H3A	120.00
N8—C1—C2	125.8 (2)	C3—C4—H4A	120.00
N8—C1—C6	117.1 (2)	C5—C4—H4A	120.00
C1—C2—C3	119.9 (3)	C4—C5—H5A	121.00
C2—C3—C4	121.0 (3)	C6—C5—H5A	121.00
C3—C4—C5	119.8 (3)	C12—C17—H22A	109.00
C4—C5—C6	118.6 (3)	C12—C17—H22B	109.00
F7—C6—C5	119.4 (2)	C18—C17—H22A	109.00
C1—C6—C5	123.6 (2)	C18—C17—H22B	109.00
F7—C6—C1	117.0 (2)	H22A—C17—H22B	108.00
N8—C9—C11	116.8 (2)	C17—C18—H21A	109.00

O10—C9—N8	120.4 (2)	C17—C18—H21B	109.00
O10—C9—C11	122.8 (2)	C19—C18—H21A	109.00
C9—C11—C12	129.8 (2)	C19—C18—H21B	109.00
C9—C11—C15	118.7 (2)	H21A—C18—H21B	108.00
C12—C11—C15	111.5 (2)	C18—C19—H20A	109.00
C11—C12—C13	113.5 (2)	C18—C19—H20B	109.00
C13—C12—C17	119.3 (2)	C20—C19—H20A	109.00
C11—C12—C17	127.1 (2)	C20—C19—H20B	109.00
S14—C13—C20	120.30 (19)	H20A—C19—H20B	108.00
C12—C13—C20	128.1 (2)	C13—C20—H18A	110.00
S14—C13—C12	111.56 (19)	C13—C20—H18B	110.00
S14—C15—C11	111.52 (19)	C19—C20—H18A	110.00
N16—C15—C11	129.6 (2)	C19—C20—H18B	110.00
S14—C15—N16	118.8 (2)	H18A—C20—H18B	108.00

C15—S14—C13—C12	−0.1 (3)	N8—C9—C11—C12	−16.9 (5)
C15—S14—C13—C20	−178.1 (3)	N8—C9—C11—C15	161.9 (3)
C13—S14—C15—N16	178.9 (3)	C9—C11—C12—C13	178.4 (3)
C13—S14—C15—C11	−0.1 (3)	C9—C11—C12—C17	−4.5 (6)
C9—N8—C1—C2	16.1 (5)	C15—C11—C12—C13	−0.4 (4)
C9—N8—C1—C6	−165.1 (3)	C15—C11—C12—C17	176.8 (3)
C1—N8—C9—O10	−7.0 (5)	C9—C11—C15—S14	−178.6 (2)
C1—N8—C9—C11	174.7 (3)	C9—C11—C15—N16	2.4 (5)
N8—C1—C2—C3	−178.1 (3)	C12—C11—C15—S14	0.3 (4)
C6—C1—C2—C3	3.2 (5)	C12—C11—C15—N16	−178.6 (3)
N8—C1—C6—F7	−1.1 (4)	C11—C12—C13—S14	0.3 (4)
N8—C1—C6—C5	179.0 (3)	C11—C12—C13—C20	178.1 (3)
C2—C1—C6—F7	177.7 (3)	C17—C12—C13—S14	−177.1 (3)
C2—C1—C6—C5	−2.1 (5)	C17—C12—C13—C20	0.7 (6)
C1—C2—C3—C4	−2.8 (6)	C11—C12—C17—C18	−160.2 (3)
C2—C3—C4—C5	1.3 (6)	C13—C12—C17—C18	16.8 (5)
C3—C4—C5—C6	−0.2 (6)	S14—C13—C20—C19	−170.8 (3)
C4—C5—C6—F7	−179.2 (3)	C12—C13—C20—C19	11.5 (5)
C4—C5—C6—C1	0.6 (6)	C12—C17—C18—C19	−47.1 (4)
O10—C9—C11—C12	164.9 (3)	C17—C18—C19—C20	61.4 (4)
O10—C9—C11—C15	−16.4 (5)	C18—C19—C20—C13	−41.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H9*A*···F7	0.86	2.26	2.643 (5)	107
N16—H15*C*···O10	0.86	2.16	2.733 (5)	124
N16—H15*D*···O10^{i}	0.86	2.25	2.986 (6)	143

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