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3-Benzylamino-2-cyano-*N*-[*N*-(2-fluorophenyl)carbamoyl]-3-(methylsulfanyl)acrylamide

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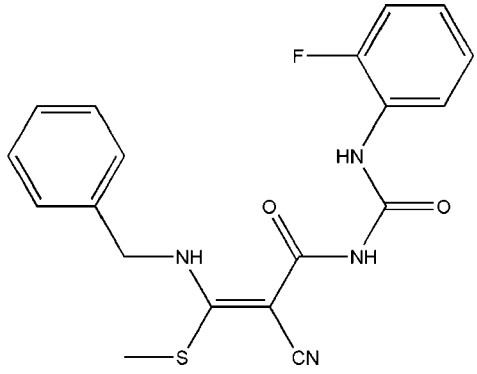
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.079; data-to-parameter ratio = 16.3.

In the crystal structure of the title compound, $C_{19}H_{17}FN_4O_2S$, molecules are linked *via* pairs of N—H···N interactions, forming centrosymmetric dimers. Two intramolecular N—H···O hydrogen bonds stabilize the molecular conformation.

Related literature

The title compound was synthesized as a herbicide. For details of the synthesis, see: Wang *et al.* (2004); Senda *et al.* (1972); Xue *et al.* (2002); Liu *et al.* (1998); Zhang *et al.* (2008).



Experimental

Crystal data

$C_{19}H_{17}FN_4O_2S$
M_r = 384.43
Triclinic, $P\overline{1}$
a = 9.2415 (18) Å
b = 10.047 (2) Å
c = 11.0949 (19) Å
α = 73.312 (6)°
β = 66.880 (6)°
γ = 84.295 (7)°
V = 907.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 133 K
0.48 × 0.39 × 0.39 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.906, T_{max} = 0.923
8644 measured reflections
4089 independent reflections
3137 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.079
S = 1.00
4089 reflections
251 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1	0.860 (17)	1.894 (17)	2.6024 (15)	138.7 (15)
N3—H3*N*···O1	0.827 (16)	1.912 (16)	2.6012 (16)	140.1 (14)
N2—H2*N*···N4^{i}	0.858 (15)	2.229 (16)	3.0710 (17)	166.8 (14)

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

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

We are grateful to Hunan Normal University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2144).

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supporting information

Acta Cryst. (2011). E**67**, o3230 [https://doi.org/10.1107/S1600536811046125]

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S1. Experimental

The title compound was prepared according to the reported method (Wang *et al.*, 2004; Senda *et al.*, 1972). Crystals of (I) suitable for XRD were obtained by slow evaporation of the acetone solution at 293 K.

S2. Refinement

Positional parameters of all H atoms were calculated geometically and were allowed to ride on the C, O and N atoms to which they are bonded, with C—H = 0.95 to 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C).

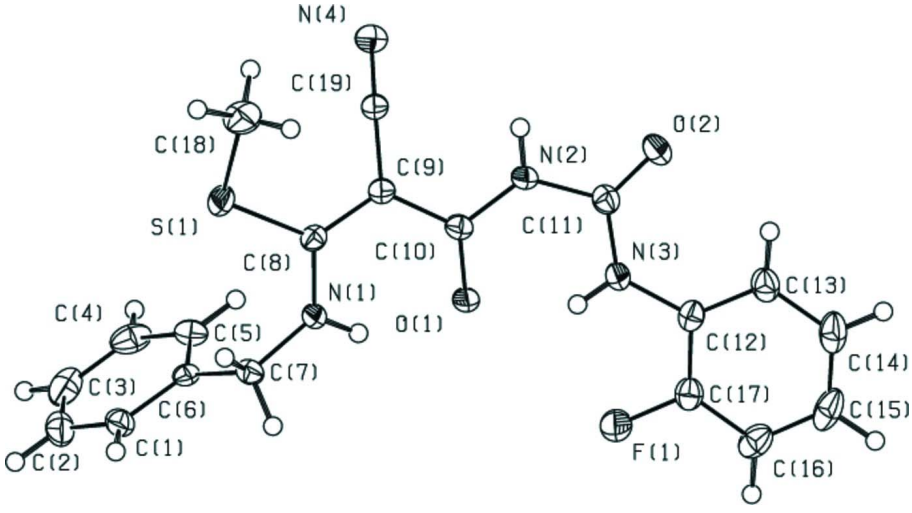

Figure 1

The molecular structure of compound shows displacement ellipsoids drawn at the 50% probability level, all H atoms have been omitted for clarity.

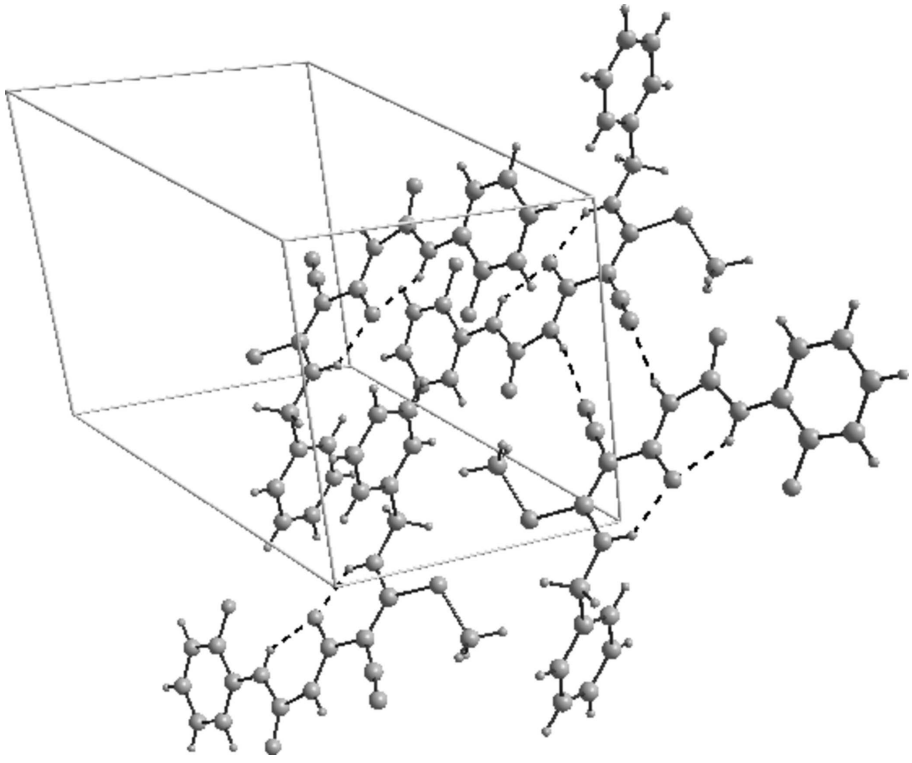

Figure 2

The crystal packing of the title compound with hydrogen bonds drawn as dashed lines.

3-Benzylamino-2-cyano-*N*-[*N*-(2- fluorophenyl)carbamoyl]-3-(methylsulfanyl)acrylamide

Crystal data

$C_{19}H_{17}FN_4O_2S$
$M_r = 384.43$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.2415\ (18)$ Å
$b = 10.047\ (2)$ Å
$c = 11.0949\ (19)$ Å
$\alpha = 73.312\ (6)°$
$\beta = 66.880\ (6)°$
$\gamma = 84.295\ (7)°$
$V = 907.4\ (3)$ Å^3
$Z = 2$
$F(000) = 400$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2908 reflections
$\theta = 2.4–29.1°$
$\mu = 0.21$ mm^{-1}
$T = 133$ K
Block, colourless
0.48 × 0.39 × 0.39 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Radiation source: Rotating Anode
Graphite monochromator
Detector resolution: 28.5714 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.906$, $T_{max} = 0.923$
8644 measured reflections
4089 independent reflections
3137 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -11\rightarrow9$
$k = -13\rightarrow13$
$l = -14\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.079$
$S = 1.00$
4089 reflections
251 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
$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2 + 0.119P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-*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 $F^2 > \sigma(F^2)$ 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_{iso}*/U_{eq}
S1	0.42534 (5)	0.14869 (4)	0.89857 (4)	0.02801 (11)
F1	0.21687 (11)	0.56288 (9)	0.25643 (8)	0.0357 (2)
O1	0.27259 (13)	0.42794 (11)	0.56187 (9)	0.0295 (3)
O2	−0.00916 (14)	0.75621 (12)	0.64645 (10)	0.0363 (3)
N1	0.43164 (14)	0.22266 (12)	0.65160 (11)	0.0222 (3)
N2	0.10887 (14)	0.55174 (12)	0.70402 (12)	0.0228 (3)
N3	0.11794 (15)	0.64303 (13)	0.48270 (12)	0.0239 (3)
N4	0.05563 (16)	0.35724 (14)	1.03305 (12)	0.0341 (3)
C1	0.63303 (18)	−0.12037 (16)	0.63660 (14)	0.0265 (3)
H1	0.7399	−0.0901	0.5950	0.032*
C2	0.5979 (2)	−0.26010 (17)	0.67324 (16)	0.0360 (4)
H2	0.6802	−0.3254	0.6567	0.043*
C3	0.4422 (2)	−0.30499 (17)	0.73435 (16)	0.0394 (4)
H3	0.4175	−0.4012	0.7596	0.047*
C4	0.3231 (2)	−0.20958 (17)	0.75847 (15)	0.0347 (4)
H4	0.2164	−0.2403	0.8006	0.042*
C5	0.35910 (18)	−0.06872 (16)	0.72123 (14)	0.0259 (3)
H5	0.2768	−0.0035	0.7382	0.031*
C6	0.51439 (16)	−0.02309 (14)	0.65956 (12)	0.0199 (3)
C7	0.56151 (16)	0.12821 (14)	0.61482 (14)	0.0223 (3)
H7A	0.6365	0.1390	0.6553	0.027*
H7B	0.6175	0.1559	0.5146	0.027*
C8	0.36183 (16)	0.25079 (14)	0.77143 (13)	0.0194 (3)
C9	0.24408 (17)	0.35141 (14)	0.79187 (13)	0.0219 (2)

C10	0.21072 (17)	0.44413 (15)	0.67827 (13)	0.0218 (3)
C11	0.06720 (17)	0.66004 (15)	0.60914 (14)	0.0235 (3)
C12	0.10494 (16)	0.73778 (15)	0.36651 (13)	0.0220 (3)
C13	0.05148 (17)	0.87350 (16)	0.35728 (15)	0.0287 (3)
H13	0.0137	0.9076	0.4355	0.034*
C14	0.05355 (18)	0.95894 (17)	0.23327 (16)	0.0354 (4)
H14	0.0189	1.0520	0.2271	0.042*
C15	0.10502 (19)	0.91076 (18)	0.11951 (16)	0.0378 (4)
H15	0.1044	0.9701	0.0359	0.045*
C16	0.15778 (19)	0.77617 (18)	0.12629 (15)	0.0330 (4)
H16	0.1927	0.7414	0.0485	0.040*
C17	0.15823 (17)	0.69412 (15)	0.24895 (14)	0.0259 (3)
C18	0.4107 (2)	0.25546 (18)	1.00866 (16)	0.0385 (4)
H18A	0.4372	0.3521	0.9544	0.058*
H18B	0.4839	0.2222	1.0548	0.058*
H18C	0.3030	0.2501	1.0767	0.058*
C19	0.14271 (17)	0.35427 (15)	0.92622 (13)	0.0219 (2)
H3N	0.1721 (19)	0.5738 (17)	0.4704 (15)	0.030 (5)*
H2N	0.0681 (19)	0.5644 (16)	0.7839 (16)	0.035 (5)*
H1N	0.394 (2)	0.2677 (18)	0.5923 (17)	0.040 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0371 (2)	0.0269 (2)	0.02340 (19)	0.00577 (17)	−0.01710 (17)	−0.00569 (15)
F1	0.0496 (6)	0.0324 (5)	0.0293 (5)	0.0090 (4)	−0.0187 (4)	−0.0121 (4)
O1	0.0418 (7)	0.0305 (6)	0.0189 (5)	0.0157 (5)	−0.0141 (5)	−0.0122 (4)
O2	0.0447 (7)	0.0339 (7)	0.0331 (6)	0.0205 (6)	−0.0174 (5)	−0.0163 (5)
N1	0.0279 (7)	0.0215 (7)	0.0191 (6)	0.0080 (5)	−0.0107 (5)	−0.0082 (5)
N2	0.0267 (7)	0.0248 (7)	0.0179 (6)	0.0071 (5)	−0.0086 (5)	−0.0096 (5)
N3	0.0289 (7)	0.0202 (7)	0.0236 (6)	0.0069 (6)	−0.0117 (5)	−0.0073 (5)
N4	0.0354 (8)	0.0392 (8)	0.0242 (7)	0.0105 (7)	−0.0087 (6)	−0.0108 (6)
C1	0.0287 (8)	0.0298 (8)	0.0253 (7)	0.0082 (7)	−0.0129 (7)	−0.0125 (6)
C2	0.0572 (12)	0.0292 (9)	0.0354 (9)	0.0155 (8)	−0.0299 (9)	−0.0166 (7)
C3	0.0736 (14)	0.0225 (9)	0.0310 (8)	−0.0057 (9)	−0.0287 (9)	−0.0059 (7)
C4	0.0418 (10)	0.0380 (10)	0.0239 (8)	−0.0138 (8)	−0.0074 (7)	−0.0103 (7)
C5	0.0257 (8)	0.0303 (8)	0.0232 (7)	0.0001 (7)	−0.0070 (6)	−0.0127 (6)
C6	0.0231 (7)	0.0241 (7)	0.0140 (6)	0.0031 (6)	−0.0079 (6)	−0.0075 (5)
C7	0.0190 (7)	0.0233 (8)	0.0216 (7)	0.0028 (6)	−0.0044 (6)	−0.0074 (6)
C8	0.0229 (7)	0.0182 (7)	0.0180 (6)	−0.0018 (6)	−0.0085 (6)	−0.0046 (5)
C9	0.0264 (5)	0.0216 (5)	0.0189 (5)	0.0030 (4)	−0.0093 (4)	−0.0073 (4)
C10	0.0241 (7)	0.0222 (7)	0.0206 (7)	0.0025 (6)	−0.0083 (6)	−0.0091 (6)
C11	0.0225 (8)	0.0234 (8)	0.0262 (7)	0.0028 (6)	−0.0108 (6)	−0.0078 (6)
C12	0.0182 (7)	0.0225 (8)	0.0243 (7)	−0.0019 (6)	−0.0099 (6)	−0.0016 (6)
C13	0.0227 (8)	0.0274 (8)	0.0339 (8)	0.0034 (6)	−0.0114 (7)	−0.0052 (6)
C14	0.0254 (8)	0.0298 (9)	0.0444 (9)	0.0018 (7)	−0.0160 (8)	0.0030 (7)
C15	0.0284 (9)	0.0434 (11)	0.0354 (9)	−0.0044 (8)	−0.0190 (8)	0.0098 (7)
C16	0.0303 (9)	0.0428 (10)	0.0262 (8)	−0.0024 (7)	−0.0146 (7)	−0.0034 (7)

C17	0.0242 (8)	0.0255 (8)	0.0291 (8)	0.0005 (6)	−0.0129 (7)	−0.0051 (6)
C18	0.0453 (10)	0.0519 (11)	0.0315 (8)	0.0081 (9)	−0.0240 (8)	−0.0198 (8)
C19	0.0264 (5)	0.0216 (5)	0.0189 (5)	0.0030 (4)	−0.0093 (4)	−0.0073 (4)

Geometric parameters (Å, º)

S1—C8	1.7567 (13)	C4—H4	0.9500
S1—C18	1.8041 (15)	C5—C6	1.385 (2)
F1—C17	1.3674 (16)	C5—H5	0.9500
O1—C10	1.2424 (16)	C6—C7	1.5085 (19)
O2—C11	1.2115 (16)	C7—H7A	0.9900
N1—C8	1.3303 (17)	C7—H7B	0.9900
N1—C7	1.4556 (17)	C8—C9	1.4060 (18)
N1—H1N	0.860 (17)	C9—C19	1.4223 (19)
N2—C10	1.3785 (17)	C9—C10	1.4577 (18)
N2—C11	1.4204 (17)	C12—C17	1.3847 (19)
N2—H2N	0.858 (15)	C12—C13	1.393 (2)
N3—C11	1.3499 (18)	C13—C14	1.391 (2)
N3—C12	1.4059 (17)	C13—H13	0.9500
N3—H3N	0.827 (16)	C14—C15	1.374 (2)
N4—C19	1.1488 (17)	C14—H14	0.9500
C1—C2	1.379 (2)	C15—C16	1.382 (2)
C1—C6	1.3903 (19)	C15—H15	0.9500
C1—H1	0.9500	C16—C17	1.375 (2)
C2—C3	1.386 (2)	C16—H16	0.9500
C2—H2	0.9500	C18—H18A	0.9800
C3—C4	1.382 (2)	C18—H18B	0.9800
C3—H3	0.9500	C18—H18C	0.9800
C4—C5	1.391 (2)		

C8—S1—C18	106.50 (7)	C9—C8—S1	124.36 (10)
C8—N1—C7	126.66 (12)	C8—C9—C19	120.11 (12)
C8—N1—H1N	113.5 (11)	C8—C9—C10	121.33 (12)
C7—N1—H1N	119.8 (11)	C19—C9—C10	118.19 (12)
C10—N2—C11	127.92 (12)	O1—C10—N2	120.88 (12)
C10—N2—H2N	120.9 (11)	O1—C10—C9	121.44 (12)
C11—N2—H2N	110.9 (11)	N2—C10—C9	117.68 (12)
C11—N3—C12	127.09 (13)	O2—C11—N3	126.13 (13)
C11—N3—H3N	116.0 (11)	O2—C11—N2	119.24 (12)
C12—N3—H3N	116.4 (11)	N3—C11—N2	114.63 (12)
C2—C1—C6	121.00 (16)	C17—C12—C13	117.29 (13)
C2—C1—H1	119.5	C17—C12—N3	116.69 (13)
C6—C1—H1	119.5	C13—C12—N3	125.92 (13)
C1—C2—C3	119.78 (16)	C14—C13—C12	119.87 (14)
C1—C2—H2	120.1	C14—C13—H13	120.1
C3—C2—H2	120.1	C12—C13—H13	120.1
C4—C3—C2	119.85 (16)	C15—C14—C13	120.97 (16)
C4—C3—H3	120.1	C15—C14—H14	119.5

C2—C3—H3	120.1	C13—C14—H14	119.5
C3—C4—C5	120.16 (16)	C14—C15—C16	120.23 (14)
C3—C4—H4	119.9	C14—C15—H15	119.9
C5—C4—H4	119.9	C16—C15—H15	119.9
C6—C5—C4	120.29 (15)	C17—C16—C15	118.02 (15)
C6—C5—H5	119.9	C17—C16—H16	121.0
C4—C5—H5	119.9	C15—C16—H16	121.0
C5—C6—C1	118.91 (14)	F1—C17—C16	119.01 (13)
C5—C6—C7	122.99 (13)	F1—C17—C12	117.38 (12)
C1—C6—C7	118.09 (13)	C16—C17—C12	123.59 (14)
N1—C7—C6	114.88 (12)	S1—C18—H18A	109.5
N1—C7—H7A	108.5	S1—C18—H18B	109.5
C6—C7—H7A	108.5	H18A—C18—H18B	109.5
N1—C7—H7B	108.5	S1—C18—H18C	109.5
C6—C7—H7B	108.5	H18A—C18—H18C	109.5
H7A—C7—H7B	107.5	H18B—C18—H18C	109.5
N1—C8—C9	121.34 (12)	N4—C19—C9	177.14 (16)
N1—C8—S1	114.28 (10)		

C6—C1—C2—C3	−0.2 (2)	C19—C9—C10—O1	−165.04 (14)
C1—C2—C3—C4	−0.1 (2)	C8—C9—C10—N2	−171.45 (13)
C2—C3—C4—C5	0.2 (2)	C19—C9—C10—N2	15.5 (2)
C3—C4—C5—C6	0.1 (2)	C12—N3—C11—O2	6.0 (3)
C4—C5—C6—C1	−0.5 (2)	C12—N3—C11—N2	−174.67 (13)
C4—C5—C6—C7	179.10 (12)	C10—N2—C11—O2	−170.90 (14)
C2—C1—C6—C5	0.52 (19)	C10—N2—C11—N3	9.7 (2)
C2—C1—C6—C7	−179.06 (12)	C11—N3—C12—C17	−176.54 (14)
C8—N1—C7—C6	81.84 (17)	C11—N3—C12—C13	7.2 (2)
C5—C6—C7—N1	4.96 (18)	C17—C12—C13—C14	−0.1 (2)
C1—C6—C7—N1	−175.49 (12)	N3—C12—C13—C14	176.14 (14)
C7—N1—C8—C9	175.63 (13)	C12—C13—C14—C15	1.2 (2)
C7—N1—C8—S1	−6.28 (19)	C13—C14—C15—C16	−0.8 (2)
C18—S1—C8—N1	148.59 (11)	C14—C15—C16—C17	−0.6 (2)
C18—S1—C8—C9	−33.39 (15)	C15—C16—C17—F1	−176.93 (14)
N1—C8—C9—C19	163.12 (14)	C15—C16—C17—C12	1.8 (2)
S1—C8—C9—C19	−14.8 (2)	C13—C12—C17—F1	177.34 (13)
N1—C8—C9—C10	−9.8 (2)	N3—C12—C17—F1	0.7 (2)
S1—C8—C9—C10	172.35 (11)	C13—C12—C17—C16	−1.4 (2)
C11—N2—C10—O1	−4.2 (2)	N3—C12—C17—C16	−178.01 (14)
C11—N2—C10—C9	175.25 (14)	C8—C9—C19—N4	−135 (3)
C8—C9—C10—O1	8.0 (2)	C10—C9—C19—N4	38 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1	0.860 (17)	1.894 (17)	2.6024 (15)	138.7 (15)

N3—H3*N*···O1	0.827 (16)	1.912 (16)	2.6012 (16)	140.1 (14)
N2—H2*N*···N4^{i}	0.858 (15)	2.229 (16)	3.0710 (17)	166.8 (14)

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