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(2*E*)-1-[2,3-Dichloro-6-methyl-5-(trifluoromethyl)phenyl]-2-(1-phenylethylidene)hydrazine

Hoong-Kun Fun,[a,b]*‡ Wan-Sin Loh,[a]§ Manjunath Bhat,[c,d] T. Arulmoli[c] and G. K. Nagaraja[d]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, PO Box 2457, Riyadh 11451, Saudi Arabia, [c]Sequent Scientific Limited, Baikampady, Karnataka, India, and [d]Department of Chemistry, Mangalore University, Mangalagangothri, Mangalore 574 199, India
Correspondence e-mail: hkfun@usm.my

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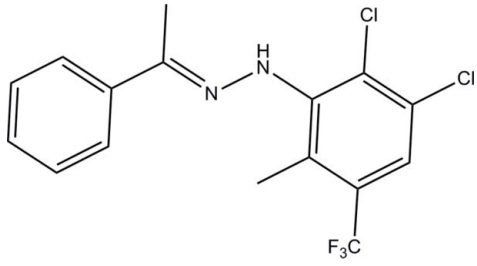
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.090; data-to-parameter ratio = 27.2.

The title compound, $C_{16}H_{13}Cl_2F_3N_2$, exists in an *E* conformation with respect to the C=N bond [1.2952 (11) Å] and the C—N—N=C torsion angle is 175.65 (8)°. The dihedral angle between the benzene rings is 42.09 (4)°. An intramolecular C—H···F hydrogen bond generates an S(6) ring. In the crystal, the molecules are linked into [101] chains by C—H···F hydrogen bonds.

Related literature

For background to the properties and applications of hydrazones, see: Barbazan *et al.* (2008); Banerjee *et al.* (2009); Ghavtadze *et al.* (2008). For related structures, see: Fun *et al.* (2011*a*,*b*). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{16}H_{13}Cl_2F_3N_2$
M_r = 361.18
Monoclinic, $P2_1/c$
a = 11.2600 (16) Å
b = 11.4025 (17) Å
c = 14.8398 (16) Å
β = 123.773 (7)°
V = 1583.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 100 K
0.32 × 0.26 × 0.22 mm

Data collection

Bruker SMART APEXII Duo CCD diffractometer
Absorption correction: multi-scan (*SADABS*); Bruker, 2009)
T_{min} = 0.872, T_{max} = 0.911
22269 measured reflections
5714 independent reflections
5043 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.090
S = 1.02
5714 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···F1^{i}	0.95	2.39	3.1652 (14)	139
C15—H15A···F2	0.98	2.38	3.1018 (13)	130

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6964).

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(2*E*)-1-[2,3-Dichloro-6-methyl-5-(trifluoromethyl)phenyl]-2-(1-phenylethylidene)hydrazine

Hoong-Kun Fun, Wan-Sin Loh, Manjunath Bhat, T. Arulmoli and G. K. Nagaraja

S1. Comment

Hydrazones are important compounds for drug design, as possible ligands for metal complexes, organocatalysis and also for the syntheses of heterocyclic compounds (e.g. Barbazan *et al.*, 2008; Banerjee *et al.*, 2009; Ghavtadze *et al.*, 2008). As part of our ongoing studies in this area (Fun *et al.*, 2011*a,b*), we now describe the structure of the title compound, (I).

The title compound, as shown in Fig. 1, exists in *trans* conformation with respect to the C7═N1 bond [C7═N1 = 1.2952 (11) Å]. An *S*(6) ring is formed *via* an intramolecular C15—H15A···F2 hydrogen bond (Table 1). The dihedral angle between the benzene rings (C1–C6 & C8–C13) is 42.09 (4)°.

In the crystal, Fig. 2, the molecules are linked into chains along [101] by C1—H1A···F1 hydrogen bonds (Table 1).

S2. Experimental

Equimolar amount of [2,3-dichloro-6-methyl-5-(trifluoromethyl)phenyl]hydrazine and acetophenone were dissolved in a minimum amount of ethanol, then followed by the addition of 0.5 ml concentrated sulfuric acid. The solution was refluxed for 8 h then cooled to room temperature and poured into ice cold water. The solid product was collected through filtration and then dried using a drying oven at 80°C. The product was redissolved in ethanol for recrystalliziation to give yellow blocks of (I). Melting point: 368 K.

S3. Refinement

N-bound H atom was located from the difference Fourier map and were refined with a riding model with U_{iso}(H) = 1.2 U_{eq}(N) [N–H = 0.8915 Å]. The remaining H atoms were positioned geometrically and refined with a riding model with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C) [C–H = 0.95 or 0.98 Å]. A rotating group model was applied to the methyl groups. In the final refinement, two outliners (1 2 2 and $\bar{1}$ 2 3) were omitted.

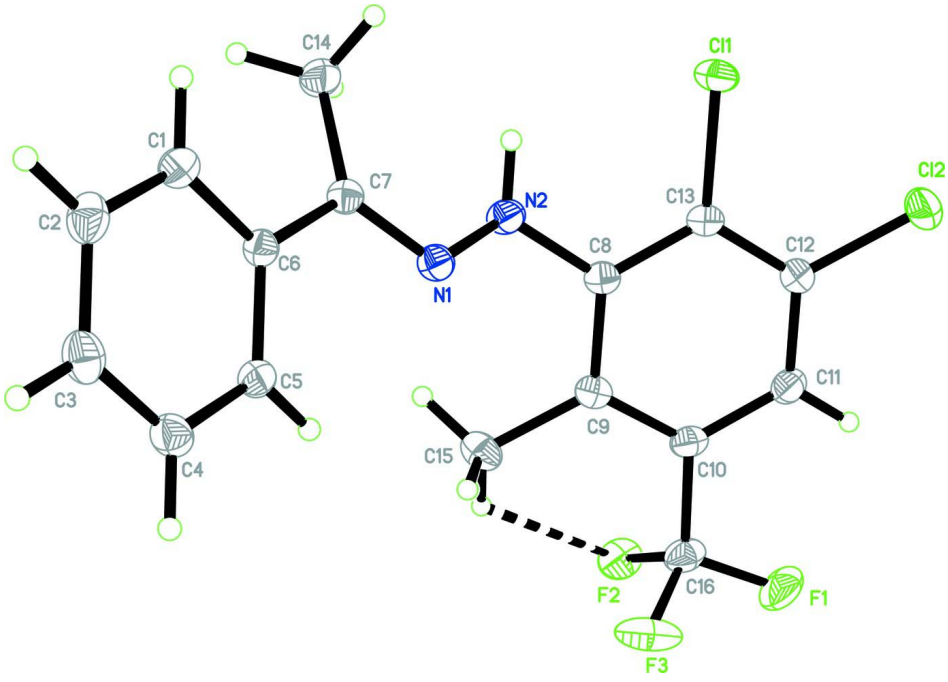

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

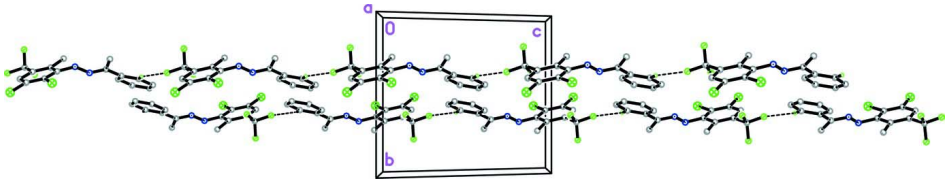

Figure 2

The crystal packing of the title compound, viewed along the *a* axis, showing the chains along [101]. H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

(2*E*)-1-[2,3-Dichloro-6-methyl-5-(trifluoromethyl)phenyl]-2- (1-phenylethylidene)hydrazine

Crystal data

$C_{16}H_{13}Cl_2F_3N_2$
$M_r = 361.18$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.2600\ (16)$ Å
$b = 11.4025\ (17)$ Å
$c = 14.8398\ (16)$ Å
$\beta = 123.773\ (7)°$
$V = 1583.8\ (4)$ Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.515$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9943 reflections
$\theta = 2.4–32.6°$
$\mu = 0.44$ mm^{-1}
$T = 100$ K
Block, yellow
0.32 × 0.26 × 0.22 mm

Data collection

Bruker SMART APEXII Duo CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans

Absorption correction: multi-scan (*SADABS*); Bruker, 2009)
T_{min} = 0.872, T_{max} = 0.911
22269 measured reflections
5714 independent reflections
5043 reflections with $I > 2\sigma(I)$

R_{int} = 0.023
θ_{max} = 32.6°, θ_{min} = 2.4°
h = −17→17
k = −17→16
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.090
S = 1.02
5714 reflections
210 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.4114P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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-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 $F^2 > \sigma(F^2)$ 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}
Cl1	0.07133 (2)	0.57463 (2)	0.919947 (18)	0.02243 (6)
Cl2	0.27412 (3)	0.51560 (3)	1.16881 (2)	0.03137 (7)
F1	0.76376 (7)	0.64100 (7)	1.26985 (6)	0.04073 (19)
F2	0.72355 (7)	0.79611 (5)	1.17471 (5)	0.02773 (13)
F3	0.76971 (7)	0.63239 (6)	1.12760 (7)	0.03906 (18)
N1	0.24945 (8)	0.64196 (7)	0.75151 (6)	0.01758 (13)
N2	0.21577 (8)	0.68019 (7)	0.82355 (6)	0.01970 (14)
H1	0.1231	0.6830	0.7982	0.024*
C1	0.08557 (10)	0.60301 (8)	0.46427 (7)	0.02097 (16)
H1A	−0.0116	0.6165	0.4387	0.025*
C2	0.11987 (11)	0.56352 (9)	0.39242 (8)	0.02343 (17)
H2A	0.0464	0.5506	0.3184	0.028*
C3	0.26177 (11)	0.54303 (8)	0.42924 (8)	0.02305 (17)
H3A	0.2855	0.5160	0.3806	0.028*
C4	0.36909 (10)	0.56246 (8)	0.53808 (8)	0.02125 (16)
H4A	0.4660	0.5484	0.5634	0.026*
C5	0.33508 (9)	0.60236 (7)	0.60973 (7)	0.01782 (15)

H5A	0.4090	0.6157	0.6836	0.021*
C6	0.19245 (9)	0.62302 (7)	0.57366 (7)	0.01647 (14)
C7	0.15569 (9)	0.66423 (7)	0.65008 (7)	0.01714 (14)
C8	0.30910 (9)	0.65100 (7)	0.93301 (7)	0.01651 (14)
C9	0.45661 (9)	0.67664 (7)	0.98943 (7)	0.01764 (15)
C10	0.54176 (9)	0.65007 (7)	1.10045 (7)	0.01872 (15)
C11	0.48613 (10)	0.60149 (8)	1.15569 (7)	0.02078 (16)
H11A	0.5468	0.5845	1.2309	0.025*
C12	0.34106 (10)	0.57840 (8)	1.09936 (7)	0.01957 (15)
C13	0.25253 (9)	0.60334 (8)	0.98898 (7)	0.01731 (14)
C14	0.01767 (10)	0.72840 (9)	0.60945 (8)	0.02298 (17)
H14A	0.0361	0.7958	0.6565	0.034*
H14B	−0.0503	0.6753	0.6102	0.034*
H14C	−0.0225	0.7558	0.5353	0.034*
C15	0.51547 (11)	0.73642 (9)	0.93086 (8)	0.02373 (17)
H15A	0.6001	0.7824	0.9828	0.036*
H15B	0.4426	0.7885	0.8747	0.036*
H15C	0.5418	0.6770	0.8972	0.036*
C16	0.69874 (10)	0.67904 (8)	1.16736 (8)	0.02398 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01517 (9)	0.02920 (11)	0.02257 (11)	−0.00304 (7)	0.01028 (8)	−0.00384 (7)
Cl2	0.02648 (12)	0.04378 (15)	0.02537 (12)	−0.00394 (10)	0.01537 (10)	0.00792 (10)
F1	0.0198 (3)	0.0430 (4)	0.0341 (4)	−0.0034 (3)	−0.0007 (3)	0.0151 (3)
F2	0.0243 (3)	0.0190 (3)	0.0311 (3)	−0.0060 (2)	0.0099 (2)	−0.0040 (2)
F3	0.0199 (3)	0.0342 (3)	0.0623 (5)	−0.0027 (2)	0.0223 (3)	−0.0143 (3)
N1	0.0180 (3)	0.0189 (3)	0.0170 (3)	0.0008 (2)	0.0105 (3)	−0.0012 (2)
N2	0.0173 (3)	0.0259 (4)	0.0161 (3)	0.0032 (3)	0.0094 (3)	−0.0008 (3)
C1	0.0184 (4)	0.0250 (4)	0.0172 (4)	−0.0010 (3)	0.0084 (3)	−0.0001 (3)
C2	0.0270 (4)	0.0244 (4)	0.0171 (4)	−0.0028 (3)	0.0111 (3)	−0.0015 (3)
C3	0.0314 (5)	0.0200 (4)	0.0232 (4)	0.0004 (3)	0.0185 (4)	−0.0007 (3)
C4	0.0228 (4)	0.0206 (4)	0.0244 (4)	0.0031 (3)	0.0157 (4)	0.0015 (3)
C5	0.0176 (3)	0.0176 (3)	0.0181 (3)	0.0016 (3)	0.0099 (3)	0.0014 (3)
C6	0.0168 (3)	0.0161 (3)	0.0162 (3)	0.0004 (3)	0.0090 (3)	0.0011 (3)
C7	0.0163 (3)	0.0177 (3)	0.0179 (3)	0.0009 (3)	0.0098 (3)	0.0005 (3)
C8	0.0161 (3)	0.0164 (3)	0.0171 (3)	0.0004 (3)	0.0092 (3)	−0.0019 (3)
C9	0.0168 (3)	0.0158 (3)	0.0209 (4)	−0.0007 (3)	0.0109 (3)	−0.0021 (3)
C10	0.0144 (3)	0.0156 (3)	0.0222 (4)	0.0002 (3)	0.0077 (3)	−0.0005 (3)
C11	0.0181 (4)	0.0196 (4)	0.0195 (4)	0.0005 (3)	0.0072 (3)	0.0025 (3)
C12	0.0185 (4)	0.0203 (4)	0.0199 (4)	−0.0006 (3)	0.0106 (3)	0.0016 (3)
C13	0.0144 (3)	0.0185 (3)	0.0183 (4)	−0.0005 (3)	0.0086 (3)	−0.0019 (3)
C14	0.0196 (4)	0.0267 (4)	0.0234 (4)	0.0066 (3)	0.0125 (3)	0.0042 (3)
C15	0.0228 (4)	0.0257 (4)	0.0258 (4)	−0.0059 (3)	0.0155 (4)	−0.0041 (3)
C16	0.0165 (4)	0.0191 (4)	0.0288 (4)	−0.0009 (3)	0.0079 (3)	0.0006 (3)

Geometric parameters (Å, º)

Cl1—C13	1.7309 (9)	C5—H5A	0.9500
Cl2—C12	1.7340 (10)	C6—C7	1.4833 (12)
F1—C16	1.3414 (12)	C7—C14	1.5075 (12)
F2—C16	1.3558 (11)	C8—C13	1.4069 (12)
F3—C16	1.3394 (13)	C8—C9	1.4139 (12)
N1—C7	1.2952 (11)	C9—C10	1.4032 (13)
N1—N2	1.3884 (10)	C9—C15	1.5165 (13)
N2—C8	1.3985 (11)	C10—C11	1.3936 (13)
N2—H1	0.8915	C10—C16	1.5060 (13)
C1—C2	1.3951 (13)	C11—C12	1.3850 (13)
C1—C6	1.4013 (12)	C11—H11A	0.9500
C1—H1A	0.9500	C12—C13	1.3936 (12)
C2—C3	1.3912 (15)	C14—H14A	0.9800
C2—H2A	0.9500	C14—H14B	0.9800
C3—C4	1.3958 (14)	C14—H14C	0.9800
C3—H3A	0.9500	C15—H15A	0.9800
C4—C5	1.3916 (12)	C15—H15B	0.9800
C4—H4A	0.9500	C15—H15C	0.9800
C5—C6	1.4020 (12)		

C7—N1—N2	115.99 (7)	C11—C10—C9	122.64 (8)
N1—N2—C8	117.97 (7)	C11—C10—C16	116.48 (8)
N1—N2—H1	116.3	C9—C10—C16	120.81 (8)
C8—N2—H1	116.5	C12—C11—C10	119.05 (8)
C2—C1—C6	120.91 (8)	C12—C11—H11A	120.5
C2—C1—H1A	119.5	C10—C11—H11A	120.5
C6—C1—H1A	119.5	C11—C12—C13	120.24 (8)
C3—C2—C1	119.96 (9)	C11—C12—Cl2	118.45 (7)
C3—C2—H2A	120.0	C13—C12—Cl2	121.31 (7)
C1—C2—H2A	120.0	C12—C13—C8	120.66 (8)
C2—C3—C4	119.63 (9)	C12—C13—Cl1	119.65 (7)
C2—C3—H3A	120.2	C8—C13—Cl1	119.69 (7)
C4—C3—H3A	120.2	C7—C14—H14A	109.5
C5—C4—C3	120.47 (9)	C7—C14—H14B	109.5
C5—C4—H4A	119.8	H14A—C14—H14B	109.5
C3—C4—H4A	119.8	C7—C14—H14C	109.5
C4—C5—C6	120.44 (8)	H14A—C14—H14C	109.5
C4—C5—H5A	119.8	H14B—C14—H14C	109.5
C6—C5—H5A	119.8	C9—C15—H15A	109.5
C1—C6—C5	118.60 (8)	C9—C15—H15B	109.5
C1—C6—C7	120.82 (8)	H15A—C15—H15B	109.5
C5—C6—C7	120.57 (7)	C9—C15—H15C	109.5
N1—C7—C6	115.64 (7)	H15A—C15—H15C	109.5
N1—C7—C14	123.57 (8)	H15B—C15—H15C	109.5
C6—C7—C14	120.78 (7)	F3—C16—F1	106.72 (9)
N2—C8—C13	118.89 (8)	F3—C16—F2	106.25 (8)

N2—C8—C9	121.03 (8)	F1—C16—F2	105.61 (8)
C13—C8—C9	119.91 (8)	F3—C16—C10	113.04 (8)
C10—C9—C8	117.49 (8)	F1—C16—C10	112.18 (8)
C10—C9—C15	122.61 (8)	F2—C16—C10	112.51 (8)
C8—C9—C15	119.82 (8)		

C7—N1—N2—C8	175.65 (8)	C15—C9—C10—C11	176.09 (8)
C6—C1—C2—C3	0.26 (14)	C8—C9—C10—C16	−177.55 (8)
C1—C2—C3—C4	−0.15 (14)	C15—C9—C10—C16	−0.72 (13)
C2—C3—C4—C5	−0.14 (14)	C9—C10—C11—C12	−0.09 (14)
C3—C4—C5—C6	0.32 (13)	C16—C10—C11—C12	176.85 (8)
C2—C1—C6—C5	−0.09 (13)	C10—C11—C12—C13	0.13 (14)
C2—C1—C6—C7	−179.58 (8)	C10—C11—C12—Cl2	178.90 (7)
C4—C5—C6—C1	−0.21 (13)	C11—C12—C13—C8	0.68 (13)
C4—C5—C6—C7	179.29 (8)	Cl2—C12—C13—C8	−178.05 (7)
N2—N1—C7—C6	−179.96 (7)	C11—C12—C13—Cl1	−179.68 (7)
N2—N1—C7—C14	0.51 (13)	Cl2—C12—C13—Cl1	1.59 (11)
C1—C6—C7—N1	157.04 (8)	N2—C8—C13—C12	−176.86 (8)
C5—C6—C7—N1	−22.44 (12)	C9—C8—C13—C12	−1.53 (13)
C1—C6—C7—C14	−23.41 (12)	N2—C8—C13—Cl1	3.50 (11)
C5—C6—C7—C14	157.11 (8)	C9—C8—C13—Cl1	178.83 (6)
N1—N2—C8—C13	−131.65 (8)	C11—C10—C16—F3	127.38 (9)
N1—N2—C8—C9	53.07 (11)	C9—C10—C16—F3	−55.61 (12)
N2—C8—C9—C10	176.75 (8)	C11—C10—C16—F1	6.65 (12)
C13—C8—C9—C10	1.52 (12)	C9—C10—C16—F1	−176.35 (8)
N2—C8—C9—C15	−0.17 (12)	C11—C10—C16—F2	−112.26 (10)
C13—C8—C9—C15	−175.40 (8)	C9—C10—C16—F2	64.74 (12)
C8—C9—C10—C11	−0.74 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···F1^{i}	0.95	2.39	3.1652 (14)	139
C15—H15*A*···F2	0.98	2.38	3.1018 (13)	130

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