Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(3-methoxyphenyl)-2,2-dimethylcyclopropanecarboxamide

Fan-Yong Yan* and Dong-Qing Liu

School of Materials Science and Chemical Engineering, Tianjin Polytechnic University, Tianjin 300160, People's Republic of China
Correspondence e-mail: yfytju@yahoo.com

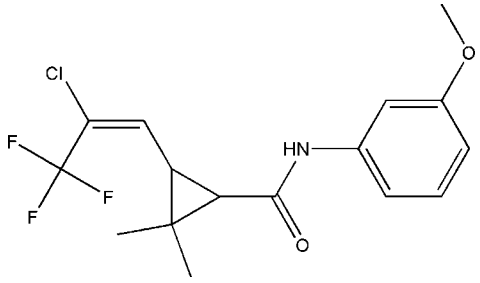
Received 20 November 2007; accepted 25 November 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.009 Å; R factor = 0.077; wR factor = 0.235; data-to-parameter ratio = 14.4.

The title compound, $C_{16}H_{17}ClF_3NO_2$, was synthesized from 3-[(*E*)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropanecarboxylic acid and 3-methoxybenzenamine. The propenyl and carboxamide substituents lie on the same side of the cyclopropane ring plane, with the two methyl substituents on either side of the plane. The benzene ring makes a dihedral angle of 76.4 (3)° with the plane of the cyclopropane ring. The crystal structure involves intermolecular N—H···O hydrogen bonds.

Related literature

For related literature, see: Liu & Yan (2007); Punja (1981).



Experimental

Crystal data

$C_{16}H_{17}ClF_3NO_2$
M_r = 347.76
Orthorhombic, *Pccn*
a = 16.785 (2) Å
b = 22.246 (3) Å
c = 9.4791 (12) Å
V = 3539.5 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 (2) K
0.22 × 0.10 × 0.01 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.947, T_{max} = 0.998
16415 measured reflections
3119 independent reflections
1325 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.236
S = 1.02
3119 reflections
216 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.901 (10)	2.046 (19)	2.928 (6)	166 (6)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation (No. 20376059) and Tianjin Polytechnic University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SF2011).

References

Bruker (1997). *SADABS*, *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, D.-Q. & Yan, F.-Y. (2007). *Acta Cryst.* E**63**, o4202.
Punja, N. (1981). European Patent EP 0031199.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supporting information

Acta Cryst. (2008). E**64**, o116 [https://doi.org/10.1107/S1600536807063167]

(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(3-methoxyphenyl)-2,2-dimethylcyclopropanecarboxamide

Fan-Yong Yan and Dong-Qing Liu

S1. Comment

We reasoned that a structure containing both of 3-((*E*)-2-Chloro- 3,3,3-trifluoroprop-1-enyl)-2,2- dimethylcyclopropanecarboxylic acid and 3-methoxybenzenamine bioactive units may show enhanced insecticidal activity and prepared the title compound (I), Fig. 1. For preparation of the title compound, see: Liu & Yan (2007); and for the insecticidal properties of related compounds, see: Punja (1981). The propenyl and carboxamide substituents lie on the same side of the C4, C5, C6 cyclopropane ring plane, with the two methyl substituents, C7 and C8 on either side of this plane. The benzene ring system is essentially planar and makes a dihedral angle of 76.4 (3)° with the plane of the cyclopropane ring. The crystal packing of (I) is shown in Fig. 2 at the end of the Comment. The packing can be described as a dimeric arrangement of molecules linked through N—H···O···H—C hydrogen bond as shown in Fig. 2 and Table 1, the packing diagram also shows F and Cl interactions..

S2. Experimental

The title compound was prepared according to the method of Liu & Yan (2007). The product was recrystallized from acetone and ethyl acetate (9:1, *v*/*v*) over 2days at ambient temperature, giving colourless single crystals of (I), (*E*)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-N– (3-methoxyphenyl)-2,2-dimethylcyclopropanecarboxamide.

S3. Refinement

H atoms were positioned geometrically with C—H = 0.93–0.98 Å and refined using riding model with U_{iso}(H) = 1.2Ueq(carrier).The N—H hydrogen atom was located in a difference Fourier map and refined freely with an isotropic displacement parameter.

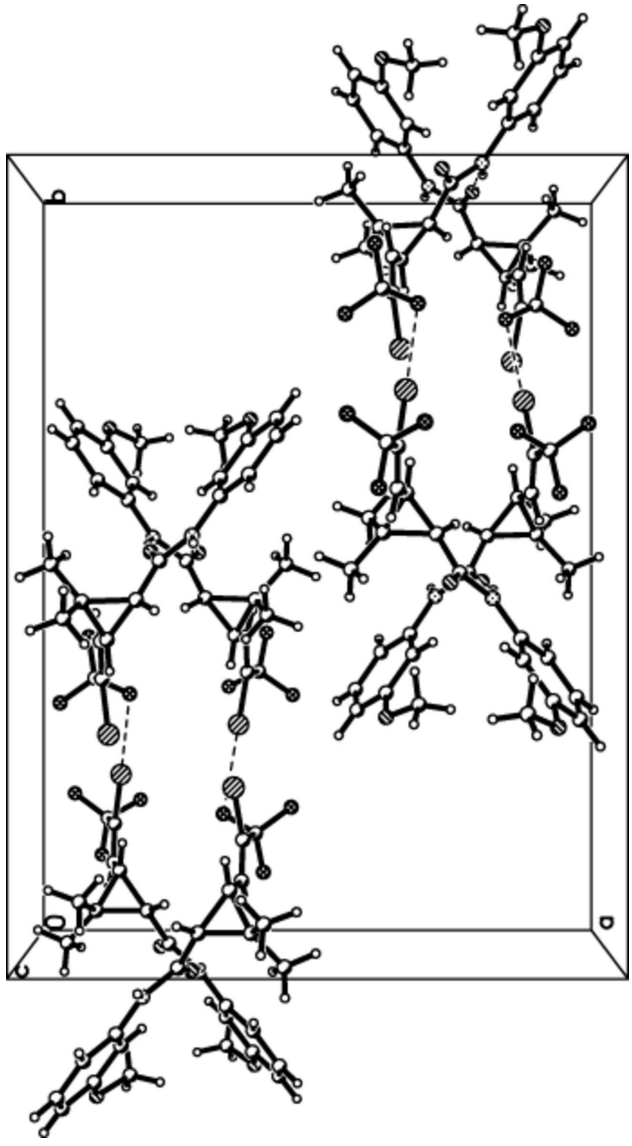


Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids.

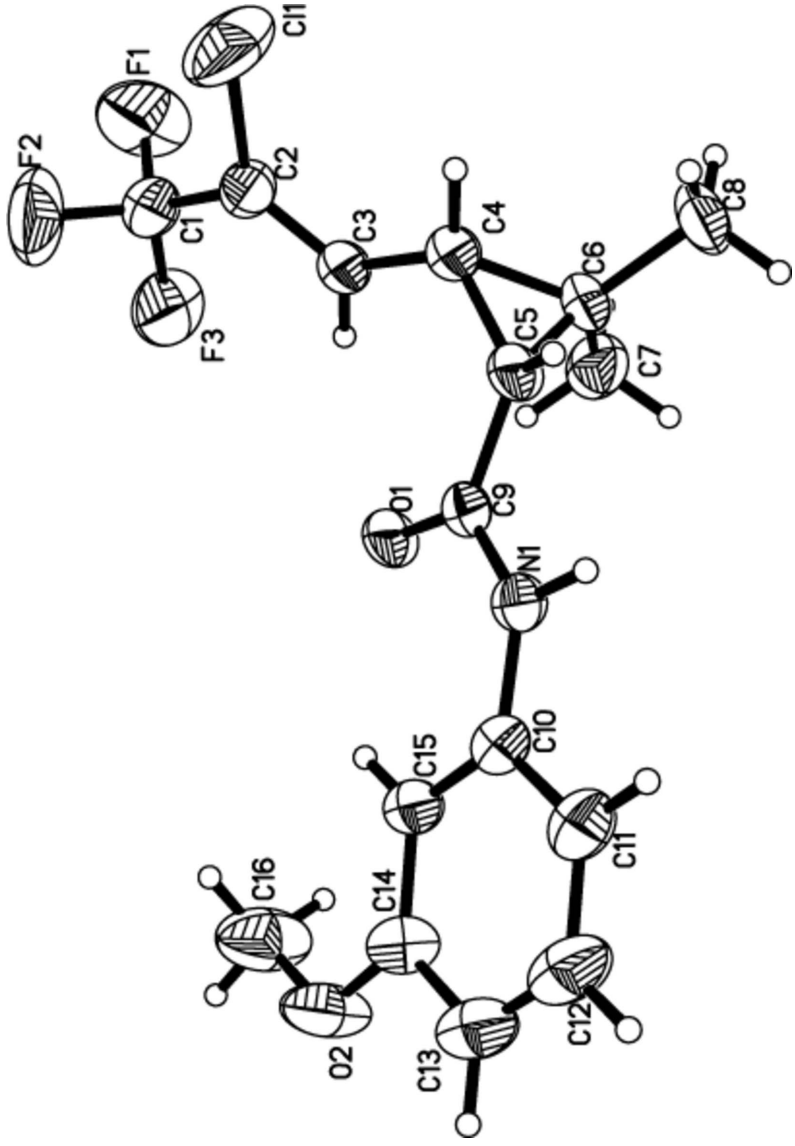


Figure 2

The crystal structure of (I), viewed along *c* axis

(*E*)-3-(2-Chloro-3,3,3-trifluoroprop-1-enyl)-*N*-(3-methoxyphenyl)-2,2- dimethylcyclopropanecarboxamide

Crystal data

$C_{16}H_{17}ClF_3NO_2$
$M_r = 347.76$
Orthorhombic, *Pccn*
$a = 16.785\ (2)$ Å
$b = 22.246\ (3)$ Å
$c = 9.4791\ (12)$ Å
$V = 3539.5\ (8)$ Å^3
$Z = 8$
$F(000) = 1440$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2222 reflections
$\theta = 2.4$–$25.9°$
$\mu = 0.25$ mm^{-1}
$T = 294$ K
Block, colourless
0.22 × 0.10 × 0.01 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.947, T_{max} = 0.998
16415 measured reflections
3119 independent reflections
1325 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 25.0°, θ_{min} = 1.5°
h = −16→19
k = −26→26
l = −11→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.236
S = 1.02
3119 reflections
216 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 13.2989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0014 (5)

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-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	0.1468 (2)	0.28489 (9)	0.5467 (3)	0.1433 (13)
C1	0.1084 (5)	0.3528 (4)	0.3245 (8)	0.085 (2)
F1	0.0466 (4)	0.3193 (3)	0.2950 (5)	0.160 (3)
F2	0.1668 (4)	0.3302 (3)	0.2454 (5)	0.155 (2)
F3	0.0957 (4)	0.4067 (2)	0.2724 (4)	0.129 (2)
O1	0.2154 (3)	0.51918 (18)	0.5554 (4)	0.0682 (12)
O2	0.3834 (4)	0.6927 (2)	0.4146 (6)	0.113 (2)
N1	0.2915 (3)	0.5393 (2)	0.7504 (5)	0.0606 (14)
C2	0.1288 (4)	0.3544 (3)	0.4765 (7)	0.0666 (18)
C3	0.1300 (4)	0.4042 (3)	0.5513 (6)	0.0613 (16)
H3	0.1190	0.4400	0.5043	0.074*
C4	0.1473 (4)	0.4082 (3)	0.7024 (6)	0.0652 (17)
H4	0.1538	0.3692	0.7492	0.078*
C5	0.1987 (4)	0.4585 (3)	0.7641 (6)	0.0635 (17)
H5	0.2319	0.4457	0.8436	0.076*

C6	0.1101 (4)	0.4567 (3)	0.7950 (6)	0.0722 (19)
C7	0.0558 (4)	0.5034 (3)	0.7319 (8)	0.091 (2)
H7A	0.0547	0.5382	0.7918	0.136*
H7B	0.0030	0.4872	0.7234	0.136*
H7C	0.0751	0.5146	0.6403	0.136*
C8	0.0874 (5)	0.4372 (4)	0.9438 (7)	0.114 (3)
H8A	0.1234	0.4065	0.9754	0.171*
H8B	0.0340	0.4217	0.9437	0.171*
H8C	0.0905	0.4711	1.0062	0.171*
C9	0.2344 (4)	0.5077 (2)	0.6786 (6)	0.0558 (15)
C10	0.3371 (4)	0.5896 (3)	0.7084 (6)	0.0598 (16)
C11	0.3868 (4)	0.6149 (3)	0.8111 (7)	0.077 (2)
H11	0.3890	0.5983	0.9011	0.092*
C12	0.4326 (5)	0.6643 (4)	0.7784 (10)	0.096 (3)
H12	0.4648	0.6815	0.8474	0.115*
C13	0.4313 (5)	0.6888 (3)	0.6451 (10)	0.092 (2)
H13	0.4636	0.7215	0.6233	0.111*
C14	0.3815 (4)	0.6643 (3)	0.5434 (8)	0.078 (2)
C15	0.3347 (4)	0.6142 (3)	0.5744 (7)	0.0675 (18)
H15	0.3022	0.5974	0.5054	0.081*
C16	0.3299 (6)	0.6743 (4)	0.3071 (9)	0.133 (4)
H16A	0.3376	0.6323	0.2878	0.199*
H16B	0.3398	0.6971	0.2230	0.199*
H16C	0.2761	0.6809	0.3378	0.199*
H1	0.297 (4)	0.529 (3)	0.842 (2)	0.09 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.222 (3)	0.0645 (13)	0.143 (2)	0.0358 (16)	−0.034 (2)	−0.0058 (13)
C1	0.108 (7)	0.078 (5)	0.071 (5)	−0.016 (5)	0.004 (5)	−0.016 (4)
F1	0.181 (6)	0.187 (6)	0.112 (4)	−0.107 (5)	−0.039 (4)	−0.009 (4)
F2	0.177 (5)	0.184 (6)	0.105 (4)	−0.016 (5)	0.042 (4)	−0.075 (4)
F3	0.212 (6)	0.118 (4)	0.055 (3)	−0.006 (4)	−0.025 (3)	0.001 (3)
O1	0.090 (3)	0.079 (3)	0.036 (2)	−0.016 (2)	−0.003 (2)	0.007 (2)
O2	0.116 (5)	0.107 (4)	0.116 (5)	−0.055 (4)	−0.012 (4)	0.031 (4)
N1	0.077 (4)	0.062 (3)	0.043 (3)	−0.008 (3)	−0.003 (3)	0.000 (3)
C2	0.076 (5)	0.061 (4)	0.063 (4)	0.003 (3)	0.000 (3)	−0.003 (3)
C3	0.077 (5)	0.059 (4)	0.048 (3)	−0.005 (3)	0.003 (3)	0.004 (3)
C4	0.086 (5)	0.059 (4)	0.051 (3)	−0.007 (4)	0.003 (3)	0.002 (3)
C5	0.084 (5)	0.070 (4)	0.037 (3)	−0.013 (4)	−0.003 (3)	0.006 (3)
C6	0.093 (5)	0.078 (5)	0.046 (3)	−0.016 (4)	0.020 (4)	−0.003 (3)
C7	0.078 (5)	0.087 (5)	0.107 (6)	−0.005 (4)	0.016 (5)	−0.022 (5)
C8	0.148 (8)	0.139 (7)	0.054 (4)	−0.057 (6)	0.037 (5)	−0.013 (5)
C9	0.070 (4)	0.056 (4)	0.042 (3)	−0.003 (3)	0.006 (3)	0.000 (3)
C10	0.061 (4)	0.057 (4)	0.061 (4)	0.000 (3)	−0.004 (3)	−0.006 (3)
C11	0.079 (5)	0.078 (5)	0.074 (5)	−0.002 (4)	−0.012 (4)	−0.014 (4)
C12	0.081 (6)	0.081 (6)	0.126 (8)	−0.012 (5)	−0.018 (5)	−0.025 (5)

C13	0.082 (6)	0.075 (5)	0.120 (7)	−0.019 (4)	−0.008 (5)	−0.005 (5)
C14	0.076 (5)	0.067 (4)	0.091 (5)	−0.012 (4)	−0.006 (4)	0.005 (4)
C15	0.078 (5)	0.062 (4)	0.063 (4)	−0.015 (4)	0.001 (3)	−0.002 (3)
C16	0.160 (9)	0.130 (8)	0.109 (7)	−0.060 (7)	−0.034 (7)	0.041 (6)

Geometric parameters (Å, º)

Cl1—C2	1.710 (6)	C6—C8	1.524 (8)
C1—F1	1.308 (8)	C7—H7A	0.9600
C1—F3	1.314 (8)	C7—H7B	0.9600
C1—F2	1.333 (9)	C7—H7C	0.9600
C1—C2	1.481 (9)	C8—H8A	0.9600
O1—C9	1.238 (6)	C8—H8B	0.9600
O2—C14	1.375 (8)	C8—H8C	0.9600
O2—C16	1.419 (9)	C10—C15	1.383 (8)
N1—C9	1.370 (7)	C10—C11	1.401 (8)
N1—C10	1.412 (7)	C11—C12	1.377 (10)
N1—H1	0.901 (10)	C11—H11	0.9300
C2—C3	1.316 (8)	C12—C13	1.377 (10)
C3—C4	1.464 (8)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.388 (9)
C4—C6	1.524 (9)	C13—H13	0.9300
C4—C5	1.530 (8)	C14—C15	1.396 (8)
C4—H4	0.9800	C15—H15	0.9300
C5—C9	1.487 (7)	C16—H16A	0.9600
C5—C6	1.516 (9)	C16—H16B	0.9600
C5—H5	0.9800	C16—H16C	0.9600
C6—C7	1.506 (9)		
F1—C1—F3	108.1 (8)	C6—C7—H7C	109.5
F1—C1—F2	104.3 (6)	H7A—C7—H7C	109.5
F3—C1—F2	104.6 (7)	H7B—C7—H7C	109.5
F1—C1—C2	113.9 (7)	C6—C8—H8A	109.5
F3—C1—C2	112.5 (6)	C6—C8—H8B	109.5
F2—C1—C2	112.8 (7)	H8A—C8—H8B	109.5
C14—O2—C16	119.3 (6)	C6—C8—H8C	109.5
C9—N1—C10	130.2 (5)	H8A—C8—H8C	109.5
C9—N1—H1	114 (4)	H8B—C8—H8C	109.5
C10—N1—H1	115 (4)	O1—C9—N1	122.8 (5)
C3—C2—C1	123.2 (6)	O1—C9—C5	124.2 (6)
C3—C2—Cl1	123.3 (5)	N1—C9—C5	113.0 (5)
C1—C2—Cl1	113.4 (5)	C15—C10—C11	119.8 (6)
C2—C3—C4	125.5 (6)	C15—C10—N1	123.8 (6)
C2—C3—H3	117.2	C11—C10—N1	116.4 (6)
C4—C3—H3	117.2	C12—C11—C10	119.7 (7)
C3—C4—C6	121.6 (6)	C12—C11—H11	120.1
C3—C4—C5	122.0 (5)	C10—C11—H11	120.1
C6—C4—C5	59.5 (4)	C13—C12—C11	121.0 (7)

C3—C4—H4	114.3	C13—C12—H12	119.5
C6—C4—H4	114.3	C11—C12—H12	119.5
C5—C4—H4	114.3	C12—C13—C14	119.4 (7)
C9—C5—C6	121.4 (6)	C12—C13—H13	120.3
C9—C5—C4	123.9 (5)	C14—C13—H13	120.3
C6—C5—C4	60.1 (4)	O2—C14—C13	115.0 (7)
C9—C5—H5	113.7	O2—C14—C15	124.6 (7)
C6—C5—H5	113.7	C13—C14—C15	120.5 (7)
C4—C5—H5	113.7	C10—C15—C14	119.5 (6)
C7—C6—C5	119.9 (6)	C10—C15—H15	120.2
C7—C6—C4	120.5 (6)	C14—C15—H15	120.2
C5—C6—C4	60.4 (4)	O2—C16—H16A	109.5
C7—C6—C8	114.4 (7)	O2—C16—H16B	109.5
C5—C6—C8	115.5 (6)	H16A—C16—H16B	109.5
C4—C6—C8	115.7 (6)	O2—C16—H16C	109.5
C6—C7—H7A	109.5	H16A—C16—H16C	109.5
C6—C7—H7B	109.5	H16B—C16—H16C	109.5
H7A—C7—H7B	109.5		

F1—C1—C2—C3	−120.4 (8)	C5—C4—C6—C8	−106.1 (7)
F3—C1—C2—C3	3.0 (11)	C10—N1—C9—O1	2.2 (10)
F2—C1—C2—C3	121.0 (8)	C10—N1—C9—C5	−178.0 (6)
F1—C1—C2—Cl1	57.3 (9)	C6—C5—C9—O1	−59.5 (8)
F3—C1—C2—Cl1	−179.3 (6)	C4—C5—C9—O1	13.4 (10)
F2—C1—C2—Cl1	−61.3 (8)	C6—C5—C9—N1	120.7 (6)
C1—C2—C3—C4	178.0 (7)	C4—C5—C9—N1	−166.4 (6)
Cl1—C2—C3—C4	0.6 (10)	C9—N1—C10—C15	−4.8 (10)
C2—C3—C4—C6	−149.2 (7)	C9—N1—C10—C11	175.1 (6)
C2—C3—C4—C5	139.4 (7)	C15—C10—C11—C12	0.5 (10)
C3—C4—C5—C9	0.8 (10)	N1—C10—C11—C12	−179.4 (6)
C6—C4—C5—C9	−109.7 (7)	C10—C11—C12—C13	−1.2 (12)
C3—C4—C5—C6	110.5 (7)	C11—C12—C13—C14	1.8 (13)
C9—C5—C6—C7	3.4 (9)	C16—O2—C14—C13	−174.8 (8)
C4—C5—C6—C7	−110.3 (7)	C16—O2—C14—C15	5.7 (12)
C9—C5—C6—C4	113.7 (6)	C12—C13—C14—O2	178.7 (7)
C9—C5—C6—C8	−139.9 (6)	C12—C13—C14—C15	−1.8 (12)
C4—C5—C6—C8	106.4 (7)	C11—C10—C15—C14	−0.5 (10)
C3—C4—C6—C7	−1.8 (9)	N1—C10—C15—C14	179.4 (6)
C5—C4—C6—C7	109.3 (7)	O2—C14—C15—C10	−179.4 (7)
C3—C4—C6—C5	−111.0 (6)	C13—C14—C15—C10	1.2 (11)
C3—C4—C6—C8	142.9 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.90 (1)	2.05 (2)	2.928 (6)	166 (6)

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