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(*Z*)-Ethyl 3-(4-chlorobenzamido)-2-cyano-3-(4-fluorophenyl)acrylate

Dehua Zhang,[a]* Xiaoyan Zhang[b] and Lijuan Guo[c]

[a]Department of Chemistry and Environmental Engineering, Hubei Normal University, Huangshi 435002, People's Republic of China, [b]School of Mathematics and Physics, Huangshi Institute of Technology, Huangshi 435003, People's Republic of China, and [c]Department of Chemistry, Changsha Medical University, Changsha 410219, People's Republic of China
Correspondence e-mail: zhangdehua200@163.com

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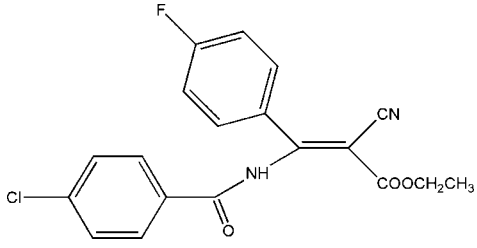
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.065; wR factor = 0.140; data-to-parameter ratio = 13.8.

The title compound, $C_{19}H_{14}ClFN_2O_3$, was prepared by the reaction of ethyl (*Z*)-3-amino-2-cyano-3-(4-fluorophenyl)-acrylate and 4-chlorobenzoyl chloride. The dihedral angle between the chlorobenzene and fluorobenzene rings is 66.18 (19)°. In addition to an intramolecular N—H···O hydrogen bond, there are intermolecular C—H···O and C—H···N hydrogen bonding interactions, which stabilize the crystal structure.

Related literature

For the agrochemical activity of the title compound, see: Heller *et al.* (2004); Ibers & Hamilton (1964).



Experimental

Crystal data

$C_{19}H_{14}ClFN_2O_3$
M_r = 372.77
Monoclinic, $P2_1/c$
a = 6.1429 (5) Å
b = 13.1555 (6) Å
c = 22.9263 (10) Å
β = 92.280 (4)°
V = 1851.27 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (2) K
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: none
23332 measured reflections
3259 independent reflections
2138 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.140
S = 1.07
3259 reflections
236 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 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···O2	0.86	2.00	2.668 (3)	134
C3—H3···N2^{i}	0.93	2.53	3.314 (5)	143
C6—H6···O1^{ii}	0.93	2.41	3.170 (4)	140
C14—H14···N2^{iii}	0.93	2.55	3.463 (5)	169

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2277).

References

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Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
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supporting information

Acta Cryst. (2009). E**65**, o90 [doi:10.1107/S1600536808041469]

(*Z*)-Ethyl 3-(4-chlorobenzamido)-2-cyano-3-(4-fluorophenyl)acrylate

Dehua Zhang, Xiaoyan Zhang and Lijuan Guo

S1. Comment

Recently, 2-cyanoacrylates have been extensively used as agrochemicals because of their unique mechanism of action and good environmental profiles. The title compound is useful as an inhibitor of Pyricularia oryzae, Rhizoctonia solani, Botrytis cinerea and Gibberella zeae (Heller *et al.*, 2004; Ibers & Hamilton, 1964).

In the title compound (Fig.1), all bond lengths and angles are unexcepional. The aromatic rings of the chlorobenzene and fluorobenzene groups form a dihedral angle of 66.18 (19)°. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond (Table 1). The crystal packing is governed by C—H···O and C—H···N hydrogen interactions (Fig.2) resulting in a three-dimensional network.

S2. Experimental

To a solution of ethyl (2*Z*)-3-amino-2-cyano-3-(4-fluorophenyl)acrylate (1.17 g, 0.0050 mol) in CH_2Cl_2 (18 ml), 4-chlorobenzoyl chloride (2.63 g, 0.015 mol) was added. Subsequently, Et_3N (1.52 g, 0.015 mol) was dropped into the solution under stirring. The reaction mixture was then heated to reflux, stirred for 4 h and cooled to room temperature. The reaction solution was filtered off and some white solid was separated. The organic phase was washed with water and then dried over Na_2SO_4. After removal of the solvent, a brown dope was obtained. The title compound was isolated by column chromatography using ethyl acetate/light petroleum (1:6 v/v) as eluent. Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature of an ethyl acetate/petroleum ether (3:1 v/v) solution after 45 days.

S3. Refinement

All H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.93-0.97 Å, N—H = 0.86 Å and with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C) for methyl H atoms.

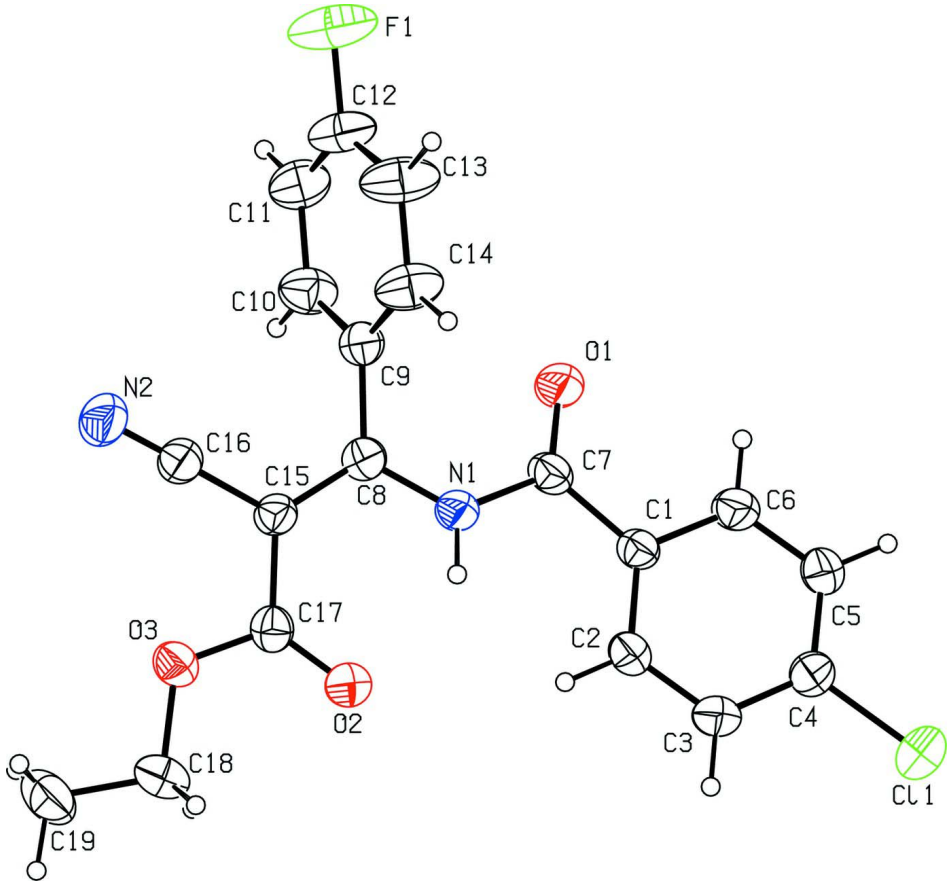

Figure 1

The molecular structure of The title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

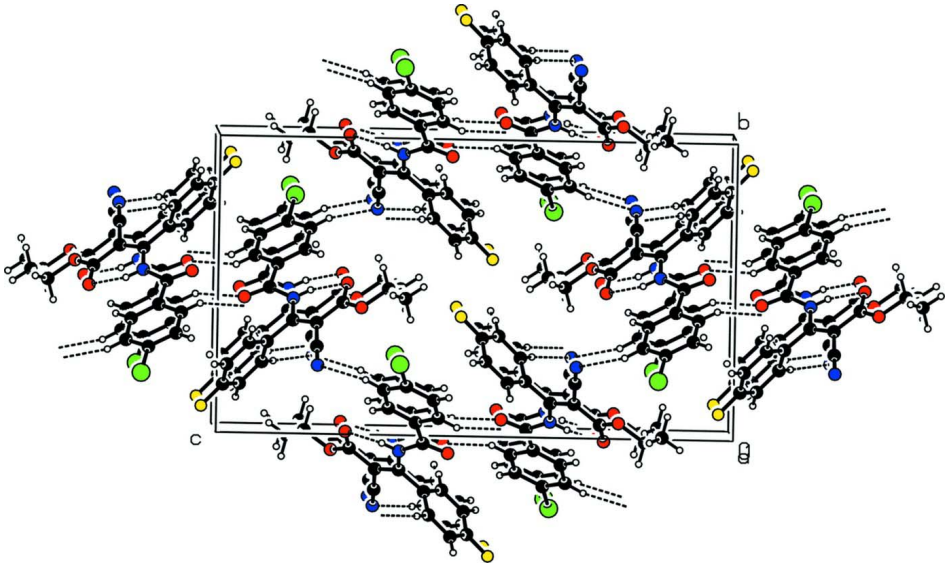

Figure 2

Packing diagram of the title compound, viewed approximately along the *a* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

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$c = 22.9263\ (10)$ Å
$\beta = 92.280\ (4)°$
$V = 1851.27\ (19)$ Å^3
$Z = 4$
$F(000) = 768$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1253 reflections
$\theta = 3.1–20.3°$
$\mu = 0.24$ mm^{-1}
$T = 298$ K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
23332 measured reflections
3259 independent reflections
2138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -7\rightarrow7$
$k = -15\rightarrow15$
$l = -24\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.140$
$S = 1.07$
3259 reflections
236 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

$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 1.3651P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.25$ 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*-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}
C1	0.4513 (5)	0.4421 (2)	0.11848 (12)	0.0510 (7)
C2	0.4752 (5)	0.3939 (2)	0.17187 (14)	0.0613 (9)
H2	0.3761	0.4064	0.2006	0.074*
C3	0.6452 (6)	0.3273 (3)	0.18272 (14)	0.0670 (9)
H3	0.6599	0.2942	0.2185	0.080*
C4	0.7924 (5)	0.3099 (2)	0.14056 (15)	0.0626 (9)
C5	0.7716 (6)	0.3559 (3)	0.08732 (15)	0.0696 (9)
H5	0.8718	0.3431	0.0589	0.084*
C6	0.6006 (6)	0.4213 (2)	0.07632 (14)	0.0673 (9)
H6	0.5848	0.4523	0.0399	0.081*
C7	0.2778 (5)	0.5173 (2)	0.10220 (14)	0.0582 (8)
C8	−0.0191 (5)	0.6171 (2)	0.14507 (13)	0.0531 (8)
C9	−0.0435 (5)	0.6899 (2)	0.09582 (13)	0.0557 (8)
C10	−0.2251 (6)	0.6897 (3)	0.05965 (16)	0.0790 (11)
H10	−0.3308	0.6400	0.0642	0.095*
C11	−0.2547 (7)	0.7618 (3)	0.01659 (16)	0.0873 (12)
H11	−0.3779	0.7598	−0.0083	0.105*
C12	−0.1070 (8)	0.8340 (3)	0.01079 (16)	0.0870 (13)
C13	0.0819 (9)	0.8367 (3)	0.04453 (19)	0.1072 (16)
H13	0.1867	0.8863	0.0388	0.129*
C14	0.1124 (7)	0.7635 (3)	0.08740 (17)	0.0879 (12)
H14	0.2395	0.7638	0.1108	0.106*
C15	−0.1571 (5)	0.6200 (2)	0.18996 (13)	0.0539 (8)
C16	−0.3251 (6)	0.6945 (3)	0.18956 (14)	0.0667 (9)
C17	−0.1424 (5)	0.5506 (2)	0.24042 (13)	0.0561 (8)
C18	−0.3118 (6)	0.4975 (3)	0.32660 (15)	0.0742 (10)
H18A	−0.1840	0.5102	0.3518	0.089*
H18B	−0.3126	0.4264	0.3155	0.089*
C19	−0.5130 (7)	0.5229 (4)	0.35748 (17)	0.1065 (15)
H19A	−0.5114	0.5937	0.3677	0.160*
H19B	−0.5193	0.4826	0.3923	0.160*
H19C	−0.6383	0.5088	0.3324	0.160*

Cl1	1.00991 (17)	0.22922 (8)	0.15591 (5)	0.0969 (4)
F1	−0.1365 (6)	0.90693 (18)	−0.03083 (11)	0.1356 (11)
N1	0.1499 (4)	0.54859 (19)	0.14698 (10)	0.0595 (7)
H1	0.1799	0.5216	0.1805	0.071*
N2	−0.4597 (6)	0.7536 (3)	0.19023 (14)	0.0981 (12)
O1	0.2500 (5)	0.5501 (2)	0.05344 (10)	0.0940 (9)
O2	0.0048 (4)	0.49015 (17)	0.24988 (10)	0.0701 (6)
O3	−0.3085 (4)	0.56200 (16)	0.27479 (9)	0.0628 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0583 (19)	0.0492 (16)	0.0460 (17)	−0.0001 (14)	0.0068 (15)	0.0007 (14)
C2	0.065 (2)	0.064 (2)	0.056 (2)	0.0058 (17)	0.0166 (17)	0.0054 (16)
C3	0.076 (2)	0.070 (2)	0.055 (2)	0.0070 (19)	0.0057 (18)	0.0142 (17)
C4	0.066 (2)	0.0532 (18)	0.069 (2)	0.0047 (16)	0.0076 (18)	0.0025 (16)
C5	0.077 (2)	0.073 (2)	0.061 (2)	0.0152 (19)	0.0198 (18)	0.0022 (18)
C6	0.085 (3)	0.066 (2)	0.0522 (19)	0.0133 (19)	0.0115 (18)	0.0070 (16)
C7	0.069 (2)	0.0603 (19)	0.0460 (19)	0.0017 (16)	0.0124 (16)	0.0019 (15)
C8	0.060 (2)	0.0509 (17)	0.0482 (18)	0.0018 (15)	−0.0006 (15)	−0.0051 (14)
C9	0.064 (2)	0.0523 (18)	0.0514 (18)	0.0036 (16)	0.0030 (16)	−0.0038 (15)
C10	0.083 (3)	0.086 (3)	0.067 (2)	−0.013 (2)	−0.011 (2)	0.013 (2)
C11	0.093 (3)	0.099 (3)	0.067 (2)	0.003 (3)	−0.025 (2)	0.008 (2)
C12	0.137 (4)	0.056 (2)	0.066 (2)	0.008 (2)	−0.021 (3)	0.0120 (19)
C13	0.150 (4)	0.066 (2)	0.102 (3)	−0.032 (3)	−0.038 (3)	0.026 (2)
C14	0.109 (3)	0.069 (2)	0.083 (3)	−0.014 (2)	−0.032 (2)	0.012 (2)
C15	0.060 (2)	0.0543 (17)	0.0471 (18)	0.0084 (15)	0.0011 (15)	−0.0003 (14)
C16	0.073 (2)	0.077 (2)	0.050 (2)	0.019 (2)	0.0033 (17)	0.0008 (17)
C17	0.057 (2)	0.0577 (19)	0.0537 (19)	0.0008 (16)	0.0027 (16)	−0.0077 (16)
C18	0.086 (3)	0.076 (2)	0.060 (2)	−0.013 (2)	0.0093 (19)	0.0138 (18)
C19	0.095 (3)	0.155 (4)	0.072 (3)	0.002 (3)	0.028 (2)	0.021 (3)
Cl1	0.0849 (7)	0.0965 (7)	0.1095 (8)	0.0329 (6)	0.0085 (6)	0.0214 (6)
F1	0.226 (3)	0.0791 (16)	0.0970 (18)	0.0063 (18)	−0.0458 (19)	0.0293 (14)
N1	0.0694 (18)	0.0653 (16)	0.0443 (14)	0.0139 (14)	0.0072 (13)	0.0055 (12)
N2	0.105 (3)	0.122 (3)	0.068 (2)	0.053 (2)	0.0133 (19)	0.0078 (19)
O1	0.110 (2)	0.119 (2)	0.0542 (15)	0.0513 (17)	0.0204 (14)	0.0236 (15)
O2	0.0746 (16)	0.0726 (14)	0.0636 (14)	0.0142 (13)	0.0100 (12)	0.0111 (12)
O3	0.0666 (14)	0.0682 (13)	0.0543 (13)	0.0006 (11)	0.0105 (11)	0.0010 (11)

Geometric parameters (Å, º)

C1—C2	1.381 (4)	C11—C12	1.324 (5)
C1—C6	1.386 (4)	C11—H11	0.9300
C1—C7	1.491 (4)	C12—F1	1.361 (4)
C2—C3	1.378 (4)	C12—C13	1.369 (6)
C2—H2	0.9300	C13—C14	1.383 (5)
C3—C4	1.369 (4)	C13—H13	0.9300
C3—H3	0.9300	C14—H14	0.9300

C4—C5	1.364 (4)	C15—C16	1.423 (5)
C4—Cl1	1.732 (3)	C15—C17	1.473 (4)
C5—C6	1.374 (4)	C16—N2	1.135 (4)
C5—H5	0.9300	C17—O2	1.217 (4)
C6—H6	0.9300	C17—O3	1.322 (4)
C7—O1	1.204 (4)	C18—O3	1.461 (4)
C7—N1	1.380 (4)	C18—C19	1.487 (5)
C8—C15	1.360 (4)	C18—H18A	0.9700
C8—N1	1.374 (4)	C18—H18B	0.9700
C8—C9	1.484 (4)	C19—H19A	0.9600
C9—C10	1.363 (4)	C19—H19B	0.9600
C9—C14	1.381 (5)	C19—H19C	0.9600
C10—C11	1.376 (5)	N1—H1	0.8600
C10—H10	0.9300		

C2—C1—C6	118.5 (3)	C11—C12—F1	120.2 (4)
C2—C1—C7	125.2 (3)	C11—C12—C13	122.1 (4)
C6—C1—C7	116.3 (3)	F1—C12—C13	117.7 (4)
C3—C2—C1	120.4 (3)	C12—C13—C14	118.1 (4)
C3—C2—H2	119.8	C12—C13—H13	121.0
C1—C2—H2	119.8	C14—C13—H13	121.0
C4—C3—C2	119.7 (3)	C9—C14—C13	120.8 (4)
C4—C3—H3	120.2	C9—C14—H14	119.6
C2—C3—H3	120.2	C13—C14—H14	119.6
C5—C4—C3	121.2 (3)	C8—C15—C16	119.2 (3)
C5—C4—Cl1	119.9 (3)	C8—C15—C17	123.8 (3)
C3—C4—Cl1	119.0 (3)	C16—C15—C17	117.0 (3)
C4—C5—C6	119.1 (3)	N2—C16—C15	178.8 (4)
C4—C5—H5	120.5	O2—C17—O3	123.6 (3)
C6—C5—H5	120.5	O2—C17—C15	124.5 (3)
C5—C6—C1	121.2 (3)	O3—C17—C15	111.9 (3)
C5—C6—H6	119.4	O3—C18—C19	107.2 (3)
C1—C6—H6	119.4	O3—C18—H18A	110.3
O1—C7—N1	121.3 (3)	C19—C18—H18A	110.3
O1—C7—C1	122.9 (3)	O3—C18—H18B	110.3
N1—C7—C1	115.7 (3)	C19—C18—H18B	110.3
C15—C8—N1	119.2 (3)	H18A—C18—H18B	108.5
C15—C8—C9	120.7 (3)	C18—C19—H19A	109.5
N1—C8—C9	120.1 (3)	C18—C19—H19B	109.5
C10—C9—C14	118.1 (3)	H19A—C19—H19B	109.5
C10—C9—C8	121.1 (3)	C18—C19—H19C	109.5
C14—C9—C8	120.6 (3)	H19A—C19—H19C	109.5
C9—C10—C11	121.1 (4)	H19B—C19—H19C	109.5
C9—C10—H10	119.4	C8—N1—C7	128.6 (3)
C11—C10—H10	119.4	C8—N1—H1	115.7
C12—C11—C10	119.7 (4)	C7—N1—H1	115.7
C12—C11—H11	120.2	C17—O3—C18	117.1 (3)
C10—C11—H11	120.2		

C6—C1—C2—C3	0.5 (5)	C10—C11—C12—C13	3.4 (7)
C7—C1—C2—C3	−178.5 (3)	C11—C12—C13—C14	−2.7 (7)
C1—C2—C3—C4	0.8 (5)	F1—C12—C13—C14	179.3 (4)
C2—C3—C4—C5	−1.3 (5)	C10—C9—C14—C13	1.9 (6)
C2—C3—C4—Cl1	177.9 (3)	C8—C9—C14—C13	−174.7 (4)
C3—C4—C5—C6	0.6 (5)	C12—C13—C14—C9	0.0 (7)
Cl1—C4—C5—C6	−178.6 (3)	N1—C8—C15—C16	176.6 (3)
C4—C5—C6—C1	0.7 (5)	C9—C8—C15—C16	−0.5 (5)
C2—C1—C6—C5	−1.3 (5)	N1—C8—C15—C17	−2.5 (5)
C7—C1—C6—C5	177.8 (3)	C9—C8—C15—C17	−179.7 (3)
C2—C1—C7—O1	−172.5 (3)	C8—C15—C17—O2	7.0 (5)
C6—C1—C7—O1	8.5 (5)	C16—C15—C17—O2	−172.2 (3)
C2—C1—C7—N1	8.1 (5)	C8—C15—C17—O3	−173.4 (3)
C6—C1—C7—N1	−170.9 (3)	C16—C15—C17—O3	7.4 (4)
C15—C8—C9—C10	−64.0 (4)	C15—C8—N1—C7	164.6 (3)
N1—C8—C9—C10	118.9 (4)	C9—C8—N1—C7	−18.2 (5)
C15—C8—C9—C14	112.5 (4)	O1—C7—N1—C8	0.0 (5)
N1—C8—C9—C14	−64.7 (4)	C1—C7—N1—C8	179.4 (3)
C14—C9—C10—C11	−1.2 (6)	O2—C17—O3—C18	−0.1 (4)
C8—C9—C10—C11	175.4 (3)	C15—C17—O3—C18	−179.7 (3)
C9—C10—C11—C12	−1.4 (6)	C19—C18—O3—C17	−179.3 (3)
C10—C11—C12—F1	−178.6 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	2.00	2.668 (3)	134
C3—H3···N2^{i}	0.93	2.53	3.314 (5)	143
C6—H6···O1^{ii}	0.93	2.41	3.170 (4)	140
C14—H14···N2^{iii}	0.93	2.55	3.463 (5)	169

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) −*x*+1, −*y*+1, −*z*; (iii) *x*+1, *y*, *z*.