



# Crystal structure of ethyl 2-(3,5-difluorophenyl)quinoline-4-carboxylate

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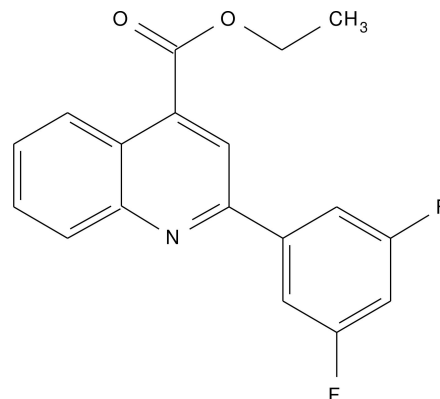
In the title compound, C<sub>18</sub>H<sub>13</sub>F<sub>2</sub>NO<sub>2</sub>, the two rings of the quinoline system are fused almost coaxially, with a dihedral angle between their planes of 2.28 (8)°. The plane of the attached benzene ring is inclined to the plane of the quinoline system by 7.65 (7)°. The carboxylate group attached to the quinoline system is in an antiperiplanar conformation. There is a short intramolecular C—H···O contact involving the carbonyl group. In the crystal, molecules are linked *via* C—H···O hydrogen bonds, forming chains lying in the (110) plane.

**Keywords:** crystal structure; quinoline derivatives; C—H···O hydrogen bonds.

**CCDC reference:** 1060299

## 1. Related literature

For the crystal structures of related quinoline derivatives, see: Pradeep *et al.* (2014); Shrunghesh Kumar *et al.* (2015).



## 2. Experimental

### 2.1. Crystal data

C<sub>18</sub>H<sub>13</sub>F<sub>2</sub>NO<sub>2</sub>  
*M<sub>r</sub>* = 313.29  
 Triclinic, *P* $\bar{1}$   
*a* = 8.2674 (3) Å  
*b* = 10.0529 (4) Å  
*c* = 10.0562 (4) Å  
 $\alpha$  = 101.193 (2)°  
 $\beta$  = 108.616 (2)°  
 $\gamma$  = 98.741 (2)°  
*V* = 756.14 (5) Å<sup>3</sup>  
*Z* = 2  
 Cu K $\alpha$  radiation  
 $\mu$  = 0.90 mm<sup>-1</sup>  
*T* = 296 K  
 0.30 × 0.27 × 0.25 mm

### 2.2. Data collection

Bruker X8 Proteum diffractometer  
 Absorption correction: multi-scan  
 (*SADABS*; Bruker, 2013)  
*T<sub>min</sub>* = 0.763, *T<sub>max</sub>* = 0.799  
 9032 measured reflections  
 2482 independent reflections  
 2147 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.038

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$  = 0.044  
 $wR(F^2)$  = 0.131  
*S* = 1.05  
 2482 reflections  
 210 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max}$  = 0.16 e Å<sup>-3</sup>  
 $\Delta\rho_{\min}$  = -0.22 e Å<sup>-3</sup>

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C6—H6···O20	0.93	2.24	2.861 (2)	123
C14—H14···O20 <sup>i</sup>	0.93	2.36	3.275 (2)	167

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); 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: SU5120).

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### References

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## supporting information

*Acta Cryst.* (2015). E71, o341–o342 [doi:10.1107/S2056989015007677]

## Crystal structure of ethyl 2-(3,5-difluorophenyl)quinoline-4-carboxylate

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### S1. Comment

Quinoline derivatives are well known for their wide range of biological and pharmaceutical activities and are prevalent in pharmacologically active natural and synthetic compounds. Quinoline-4-carboxylates are potential 5HT<sub>3</sub> antagonist and also they possess anti-emetic activity. In view of their broad spectrum of medicinal properties and in continuation of our work on new quinoline-based therapeutic agents (Pradeep *et al.*, 2014, Shringesh Kumar *et al.*, 2015), we have synthesized the title compound and report herein on its crystal structure.

The structure of the compound is shown in Fig. 1. The dihedral angle between the benzene ring and the quinoline ring system is 7.65 (7)°. The carboxylate group attached to the quinoline moiety is in an *-anti periplanar* conformation which is evident by the torsion angle C22—O21—C19—C8 = -176.71 (15)°. The two rings of the quinoline moiety are fused in an axial fashion with a dihedral angle value of 2.28 (8)°. The deviation of the bond length values for C8—C19 and C10—C11 from the standard values can be attributed for the *sp*<sup>3</sup> hybridization.

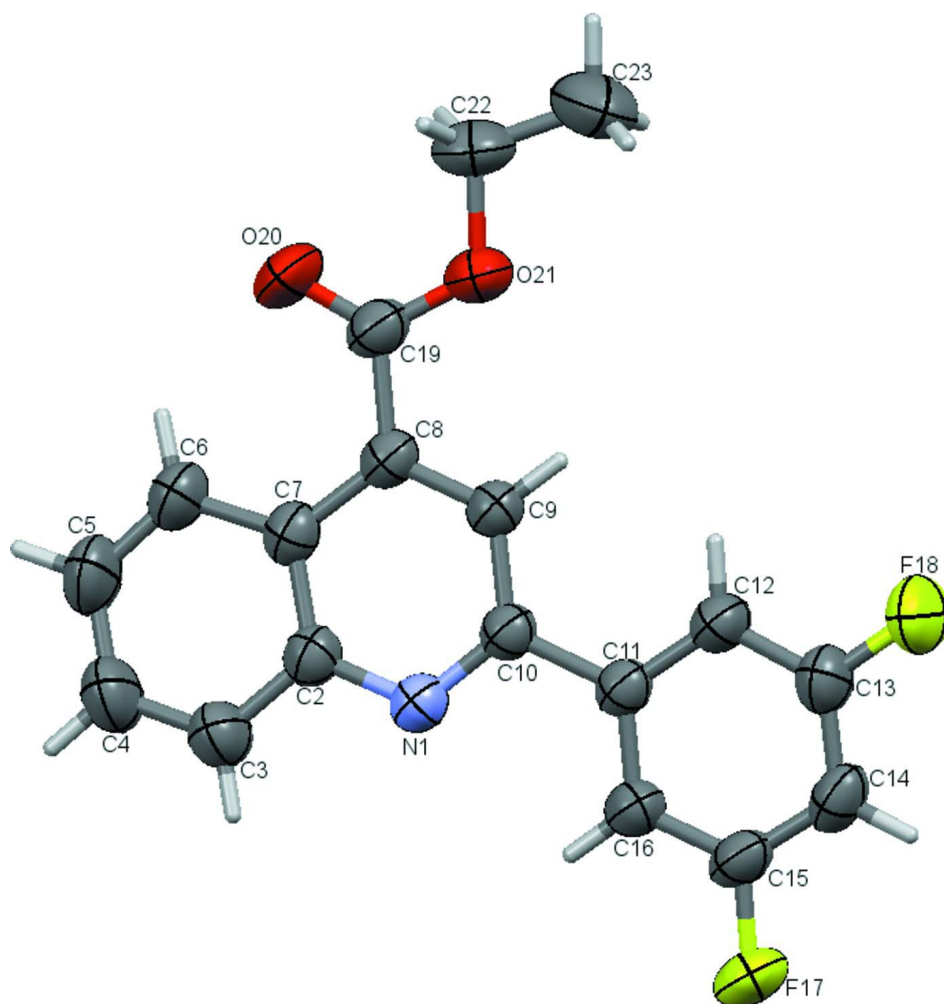
In the crystal, molecules are linked via C—H···O hydrogen bonds forming chains lying in the (1 $\bar{1}$ 0) plane. (Fig. 2 and Table 1).

### S2. Synthesis and crystallization

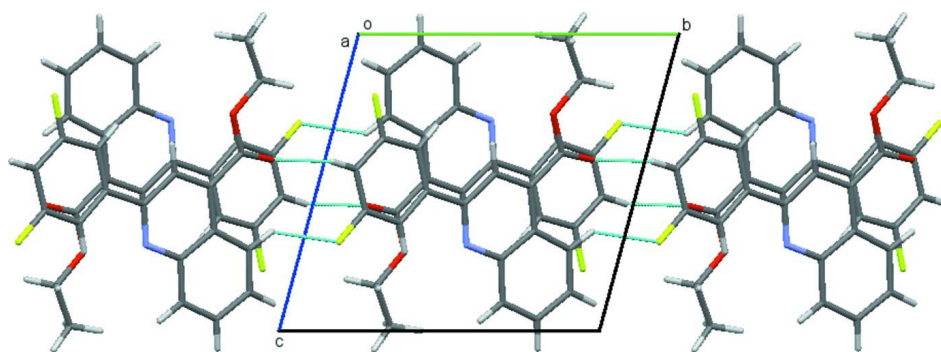
To a solution of 2-(3,5-difluorophenyl)quinoline-4-carboxylic acid (0.5 g) in 20 ml of EtOH, 1 ml of H<sub>2</sub>SO<sub>4</sub> (conc.) was added. The resulting reaction mixture was refluxed for 15 h. Solvent was removed under reduced pressure and the residue was partitioned between EtOAc and saturated NaHCO<sub>3</sub> solution. The organic layer was washed with water and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and condensed to give the title compound as a white solid (yield: 93%). The compound was dissolved in dimethylformamide and the solution was gently heated and left undisturbed. Brown, rectangular crystals grew after 12 days by slow evaporation of the solvent.

### S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All the H atoms were fixed geometrically (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl H atoms and  $1.2U_{\text{eq}}(\text{C})$  for other H atoms.

**Figure 1**

View of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details).

## Ethyl 2-(3,5-difluorophenyl)quinoline-4-carboxylate

## Crystal data

C<sub>18</sub>H<sub>13</sub>F<sub>2</sub>NO<sub>2</sub> $M_r = 313.29$ Triclinic,  $P\bar{1}$ 

Hall symbol: -P 1

 $a = 8.2674 (3) \text{ \AA}$  $b = 10.0529 (4) \text{ \AA}$  $c = 10.0562 (4) \text{ \AA}$  $\alpha = 101.193 (2)^\circ$  $\beta = 108.616 (2)^\circ$  $\gamma = 98.741 (2)^\circ$  $V = 756.14 (5) \text{ \AA}^3$  $Z = 2$  $F(000) = 324$  $D_x = 1.376 \text{ Mg m}^{-3}$ Cu  $K\alpha$  radiation,  $\lambda = 1.54178 \text{ \AA}$ 

Cell parameters from 2482 reflections

 $\theta = 6.1\text{--}64.4^\circ$  $\mu = 0.90 \text{ mm}^{-1}$  $T = 296 \text{ K}$ 

Rectangle, brown

 $0.30 \times 0.27 \times 0.25 \text{ mm}$ 

## Data collection

Bruker X8 Proteum

diffractometer

Radiation source: Bruker MicroStar microfocus

rotating anode

Helios multilayer optics monochromator

Detector resolution: 10.7 pixels  $\text{mm}^{-1}$  $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

 $T_{\min} = 0.763$ ,  $T_{\max} = 0.799$ 

9032 measured reflections

2482 independent reflections

2147 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.038$  $\theta_{\max} = 64.4^\circ$ ,  $\theta_{\min} = 6.1^\circ$  $h = -9 \rightarrow 9$  $k = -11 \rightarrow 11$  $l = -11 \rightarrow 11$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.044$  $wR(F^2) = 0.131$  $S = 1.05$ 

2482 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

 $w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.099P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$  $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$ 

Extinction correction: SHELXL97 (Sheldrick,

2008),  $FC^* = KFC[1 + 0.001XFC^2\Lambda^3/\text{SIN}(2\Theta)]^{-1/4}$ 

Extinction coefficient: 0.011 (3)

## 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
F17	-0.07505 (13)	0.11215 (10)	0.69978 (11)	0.0832 (4)

F18	-0.21878 (16)	0.10400 (12)	0.20982 (12)	0.0993 (4)
O20	0.5498 (2)	0.83249 (15)	0.42261 (16)	0.1012 (6)
O21	0.31565 (15)	0.67459 (11)	0.26915 (12)	0.0665 (4)
N1	0.35974 (15)	0.51312 (12)	0.70955 (13)	0.0525 (4)
C2	0.48895 (19)	0.63200 (14)	0.75527 (16)	0.0520 (4)
C3	0.5941 (2)	0.67423 (17)	0.90376 (18)	0.0698 (6)
C4	0.7210 (3)	0.7948 (2)	0.9594 (2)	0.0830 (7)
C5	0.7486 (3)	0.87609 (19)	0.8681 (2)	0.0837 (7)
C6	0.6535 (2)	0.83789 (16)	0.72399 (19)	0.0682 (6)
C7	0.51962 (18)	0.71364 (14)	0.66138 (16)	0.0512 (5)
C8	0.41001 (18)	0.66272 (14)	0.51179 (16)	0.0477 (4)
C9	0.28096 (18)	0.54385 (14)	0.46942 (15)	0.0475 (4)
C10	0.25718 (17)	0.47149 (13)	0.57158 (15)	0.0458 (4)
C11	0.11409 (17)	0.34387 (13)	0.52808 (15)	0.0473 (4)
C12	0.0105 (2)	0.28135 (15)	0.38426 (17)	0.0578 (5)
C13	-0.1193 (2)	0.16400 (16)	0.35114 (17)	0.0622 (5)
C14	-0.1529 (2)	0.10394 (15)	0.45354 (19)	0.0610 (5)
C15	-0.0474 (2)	0.16870 (15)	0.59420 (18)	0.0577 (5)
C16	0.08370 (19)	0.28567 (15)	0.63472 (17)	0.0544 (5)
C19	0.4343 (2)	0.73427 (15)	0.39981 (18)	0.0573 (5)
C22	0.3365 (3)	0.7336 (2)	0.1529 (2)	0.0850 (8)
C23	0.1898 (4)	0.6564 (3)	0.0175 (3)	0.1138 (10)
H3	0.57680	0.61930	0.96470	0.0840*
H4	0.78890	0.82250	1.05790	0.1000*
H5	0.83450	0.95880	0.90700	0.1000*
H6	0.67630	0.89370	0.66540	0.0820*
H9	0.20810	0.51030	0.37230	0.0570*
H12	0.02840	0.31830	0.31080	0.0690*
H14	-0.24150	0.02450	0.42900	0.0730*
H16	0.15150	0.32560	0.73220	0.0650*
H22A	0.44750	0.72490	0.14270	0.1020*
H22B	0.33500	0.83160	0.17440	0.1020*
H23A	0.18670	0.55860	0.00140	0.1710*
H23B	0.20580	0.68790	-0.06260	0.1710*
H23C	0.08140	0.67240	0.02550	0.1710*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
F17	0.0858 (7)	0.0796 (7)	0.0865 (7)	-0.0067 (5)	0.0321 (6)	0.0446 (5)
F18	0.1077 (9)	0.0813 (7)	0.0655 (7)	-0.0309 (6)	0.0073 (6)	0.0027 (5)
O20	0.1060 (11)	0.0881 (9)	0.0877 (10)	-0.0363 (8)	0.0244 (8)	0.0372 (7)
O21	0.0731 (7)	0.0690 (7)	0.0589 (7)	0.0017 (5)	0.0253 (6)	0.0284 (5)
N1	0.0544 (7)	0.0478 (7)	0.0520 (7)	0.0038 (5)	0.0171 (6)	0.0154 (5)
C2	0.0507 (8)	0.0465 (7)	0.0545 (8)	0.0059 (6)	0.0162 (7)	0.0122 (6)
C3	0.0755 (11)	0.0633 (10)	0.0568 (9)	0.0024 (8)	0.0119 (8)	0.0151 (7)
C4	0.0820 (12)	0.0736 (11)	0.0620 (11)	-0.0057 (9)	0.0003 (9)	0.0091 (9)
C5	0.0757 (12)	0.0615 (10)	0.0820 (13)	-0.0157 (8)	0.0065 (10)	0.0086 (9)

C6	0.0654 (10)	0.0546 (9)	0.0720 (11)	-0.0056 (7)	0.0173 (8)	0.0156 (8)
C7	0.0478 (8)	0.0445 (7)	0.0587 (9)	0.0066 (6)	0.0186 (7)	0.0118 (6)
C8	0.0463 (7)	0.0436 (7)	0.0572 (8)	0.0086 (6)	0.0225 (6)	0.0168 (6)
C9	0.0464 (7)	0.0455 (7)	0.0497 (8)	0.0064 (6)	0.0166 (6)	0.0145 (6)
C10	0.0452 (7)	0.0428 (7)	0.0503 (8)	0.0082 (6)	0.0178 (6)	0.0145 (6)
C11	0.0454 (7)	0.0431 (7)	0.0547 (8)	0.0080 (6)	0.0187 (6)	0.0163 (6)
C12	0.0628 (9)	0.0505 (8)	0.0564 (9)	0.0002 (7)	0.0208 (7)	0.0169 (7)
C13	0.0615 (9)	0.0513 (8)	0.0595 (9)	-0.0003 (7)	0.0129 (7)	0.0079 (7)
C14	0.0540 (9)	0.0432 (8)	0.0819 (11)	0.0004 (6)	0.0227 (8)	0.0185 (7)
C15	0.0561 (9)	0.0513 (8)	0.0731 (10)	0.0075 (6)	0.0270 (8)	0.0303 (7)
C16	0.0525 (8)	0.0534 (8)	0.0567 (9)	0.0053 (6)	0.0175 (7)	0.0217 (7)
C19	0.0578 (9)	0.0518 (8)	0.0661 (10)	0.0065 (7)	0.0258 (8)	0.0227 (7)
C22	0.0999 (14)	0.0970 (14)	0.0718 (12)	0.0123 (11)	0.0406 (11)	0.0442 (10)
C23	0.1130 (18)	0.156 (2)	0.0710 (13)	0.0141 (16)	0.0238 (12)	0.0552 (14)

*Geometric parameters (Å, °)*

F17—C15	1.3611 (19)	C11—C16	1.388 (2)
F18—C13	1.3528 (19)	C12—C13	1.374 (2)
O20—C19	1.194 (2)	C13—C14	1.369 (2)
O21—C19	1.319 (2)	C14—C15	1.367 (2)
O21—C22	1.454 (2)	C15—C16	1.367 (2)
N1—C2	1.366 (2)	C22—C23	1.475 (4)
N1—C10	1.3171 (18)	C3—H3	0.9300
C2—C3	1.407 (2)	C4—H4	0.9300
C2—C7	1.421 (2)	C5—H5	0.9300
C3—C4	1.363 (3)	C6—H6	0.9300
C4—C5	1.390 (3)	C9—H9	0.9300
C5—C6	1.353 (3)	C12—H12	0.9300
C6—C7	1.418 (2)	C14—H14	0.9300
C7—C8	1.427 (2)	C16—H16	0.9300
C8—C9	1.368 (2)	C22—H22A	0.9700
C8—C19	1.497 (2)	C22—H22B	0.9700
C9—C10	1.414 (2)	C23—H23A	0.9600
C10—C11	1.492 (2)	C23—H23B	0.9600
C11—C12	1.383 (2)	C23—H23C	0.9600
C19—O21—C22	115.85 (14)	O20—C19—O21	122.45 (16)
C2—N1—C10	118.74 (12)	O20—C19—C8	124.87 (16)
N1—C2—C3	117.08 (14)	O21—C19—C8	112.65 (14)
N1—C2—C7	123.41 (13)	O21—C22—C23	107.77 (19)
C3—C2—C7	119.51 (14)	C2—C3—H3	120.00
C2—C3—C4	120.79 (16)	C4—C3—H3	120.00
C3—C4—C5	119.67 (17)	C3—C4—H4	120.00
C4—C5—C6	121.59 (19)	C5—C4—H4	120.00
C5—C6—C7	120.72 (16)	C4—C5—H5	119.00
C2—C7—C6	117.68 (14)	C6—C5—H5	119.00
C2—C7—C8	116.27 (13)	C5—C6—H6	120.00

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C6—C7—C8	126.04 (14)	C7—C6—H6	120.00
C7—C8—C9	118.95 (13)	C8—C9—H9	120.00
C7—C8—C19	121.86 (13)	C10—C9—H9	120.00
C9—C8—C19	119.19 (13)	C11—C12—H12	120.00
C8—C9—C10	120.74 (13)	C13—C12—H12	120.00
N1—C10—C9	121.83 (13)	C13—C14—H14	122.00
N1—C10—C11	116.69 (12)	C15—C14—H14	122.00
C9—C10—C11	121.48 (12)	C11—C16—H16	121.00
C10—C11—C12	121.97 (13)	C15—C16—H16	121.00
C10—C11—C16	119.24 (13)	O21—C22—H22A	110.00
C12—C11—C16	118.79 (14)	O21—C22—H22B	110.00
C11—C12—C13	119.22 (14)	C23—C22—H22A	110.00
F18—C13—C12	118.35 (14)	C23—C22—H22B	110.00
F18—C13—C14	118.13 (15)	H22A—C22—H22B	108.00
C12—C13—C14	123.52 (15)	C22—C23—H23A	109.00
C13—C14—C15	115.41 (15)	C22—C23—H23B	109.00
F17—C15—C14	117.53 (14)	C22—C23—H23C	110.00
F17—C15—C16	118.38 (14)	H23A—C23—H23B	109.00
C14—C15—C16	124.09 (15)	H23A—C23—H23C	109.00
C11—C16—C15	118.97 (14)	H23B—C23—H23C	109.00
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C19—O21—C22—C23	-178.32 (19)	C19—C8—C9—C10	178.34 (14)
C22—O21—C19—O20	1.5 (3)	C9—C8—C19—O21	3.7 (2)
C22—O21—C19—C8	-176.71 (15)	C7—C8—C19—O20	4.4 (3)
C10—N1—C2—C7	-0.3 (2)	C7—C8—C19—O21	-177.37 (14)
C10—N1—C2—C3	179.08 (15)	C9—C8—C19—O20	-174.49 (18)
C2—N1—C10—C9	2.1 (2)	C8—C9—C10—N1	-1.7 (2)
C2—N1—C10—C11	-177.91 (13)	C8—C9—C10—C11	178.32 (14)
N1—C2—C3—C4	-177.18 (18)	N1—C10—C11—C12	-172.73 (14)
C3—C2—C7—C6	-1.9 (2)	C9—C10—C11—C16	-172.80 (14)
C3—C2—C7—C8	178.80 (15)	N1—C10—C11—C16	7.2 (2)
N1—C2—C7—C6	177.45 (15)	C9—C10—C11—C12	7.3 (2)
N1—C2—C7—C8	-1.9 (2)	C16—C11—C12—C13	0.1 (2)
C7—C2—C3—C4	2.2 (3)	C10—C11—C12—C13	-179.96 (16)
C2—C3—C4—C5	-0.9 (3)	C10—C11—C16—C15	-179.93 (15)
C3—C4—C5—C6	-0.7 (4)	C12—C11—C16—C15	0.0 (2)
C4—C5—C6—C7	1.0 (3)	C11—C12—C13—F18	-179.73 (15)
C5—C6—C7—C2	0.3 (3)	C11—C12—C13—C14	-0.1 (3)
C5—C6—C7—C8	179.57 (18)	C12—C13—C14—C15	-0.1 (3)
C6—C7—C8—C9	-177.03 (16)	F18—C13—C14—C15	179.58 (15)
C2—C7—C8—C19	-176.71 (14)	C13—C14—C15—F17	-179.53 (15)
C2—C7—C8—C9	2.2 (2)	C13—C14—C15—C16	0.2 (3)
C6—C7—C8—C19	4.0 (3)	F17—C15—C16—C11	179.57 (14)
C7—C8—C9—C10	-0.6 (2)	C14—C15—C16—C11	-0.2 (3)

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*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C6—H6 $\cdots$ O20	0.93	2.24	2.861 (2)	123
C14—H14 $\cdots$ O20 <sup>i</sup>	0.93	2.36	3.275 (2)	167

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

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