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3-(4-Fluorophenyl)imidazo[1,2-*a*]pyridine-2-carbaldehyde

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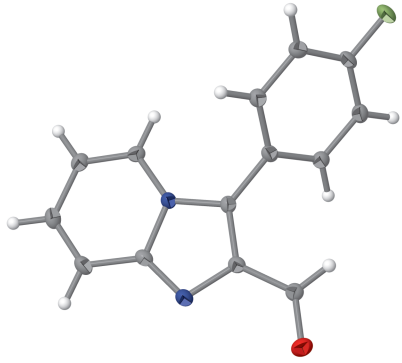
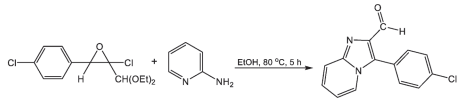
In the title compound, $C_{14}H_9FN_2O$, the dihedral angle between the imidazo–pyridine fused ring system and the pendant fluorophenyl ring is 53.77 (4)°. In the crystal, C—H···O and C—H···F hydrogen bonds link the molecules into a three-dimensional network. A Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H···H (30.4%), H···C/C···H (23.7%), H···O/O···H (12.2%) and H···F/F···H (11.1%) interactions.

3D view



Chemical scheme



Structure description

Imidazo[1,2-*a*]pyridines are an important class of fused N-bridged compounds because of the broad spectrum of synthetic transformations as well as biological activity profiles displayed, which are strongly affected by the substitutions. Several imidazo[1,2-*a*]pyridines are used clinically, such as the unsubstituted imidazole skeleton cardiotonic agent olprinone, the anticancer agent zolimidine, the 2-substituted analgesic miroprofen, the 3-substituted antiosteoporosis 2,3-disubstituted derivatives with anxiolytic and sedative properties, saripidem, alpidem, and necopidem, and the agent for the treatment of brain disorders and insomnia, zolpidem (Kurteva, 2021). As part of our ongoing studies in this area, we now report the synthesis and structure of the title compound (**I**) (Fig. 1).

The dihedral angle between the N1/N2/C2/C3/C5–C8/C8*A* fused ring system (r.m.s. deviation = 0.015 Å) and the pendant C11–C16 fluorobenzene ring is 53.77 (4)°. The aldehyde O atom lies close to the plane of the fused ring as indicted by the

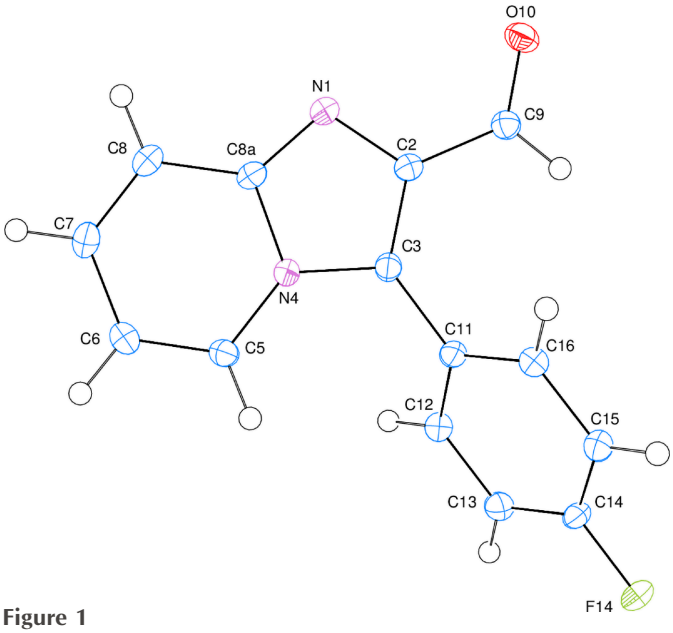


Figure 1
The molecular structure of (**I**) showing 50% probability displacement ellipsoids.

N1—C2—C9═O10 torsion angle of 4.15 (17)°. Atom F14 lies −0.0118 (8) Å away from the best least-squares plane of the phenyl ring.

In the crystal, C—H···O and C—H···F hydrogen bonds (Table 1) link the molecules into a three-dimensional network, enclosing $R_2^2(14)$, $R_3^3(16)$, $R_4^4(18)$, $R_4^4(24)$ and $R_4^4(30)$ ring motifs (Fig. 2). It may be noted that the aldehyde O10 atom accepts three such bonds in a distorted tetrahedral arrangement (including the C8═O10 bond). Atom F14 accepts two hydrogen bonds in a distorted trigonal arrangement including the C14—F1 bond. Further, there is a weak π–π stacking interaction between the pyridine rings with a centroid–centroid distance of 3.9090 (7) Å. No significant C—H···π interactions are observed.

In order to further visualize the intermolecular interactions in the crystal of (**I**), a Hirshfeld surface analysis (Fig. 3) was carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021). The overall two-dimensional fingerprint plot, Fig. 4*a*, and those delineated into different contact types (McKinnon *et al.*, 2007) are illustrated in Fig. 4*b–l*. These indicate that the most important contributions to the crystal packing are from H···H (30.4%), H···C/C···H (23.7%), H···O/O···H (12.2%) and H···F/F···H (11.1%) interactions.

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O10^{i}	0.95	2.45	3.3579 (14)	160
C12—H12···O10^{ii}	0.95	2.45	3.3449 (15)	158
C16—H16···O10^{iii}	0.95	2.47	3.3754 (14)	159
C5—H5···F14^{iv}	0.95	2.50	3.1435 (13)	125
C9—H9···F14^{v}	0.95	2.55	3.2089 (14)	127

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Synthesis and crystallization

A solution of equimolar amounts of 2-aminopyridine (2 mmol) and 2-chloro-2-(diethoxymethyl)-3-(4-fluorophenyl) oxirane (2 mmol) in 20 ml of 95% aqueous ethanol was heated at reflux for 5 h. The solvent was removed *in vacuo*, and the remaining white powder was recrystallized from dry acetonitrile solution to give the title compound in the form of colorless prisms. Yield: 45%, m.p. 420–421 K. Analysis calculated for $C_{14}H_9FN_2O$: C, 69.99; H, 3.78; N, 11.66. Found: C, 69.96; H, 3.75; N, 11.63. ^{1}H NMR (300 MHz, DMSO-d_6): 6.86–8.47 (8*H*, Ar) and 9.79 (1*H*, CHO). ^{13}C NMR (200 MHz, DMSO-d_6): 111.89, 115.78, 116.90, 111.87, 121.32, 126.18, 130.39, 132.79, 140.89, 147.48, 160.87, 165.96, 185.45.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

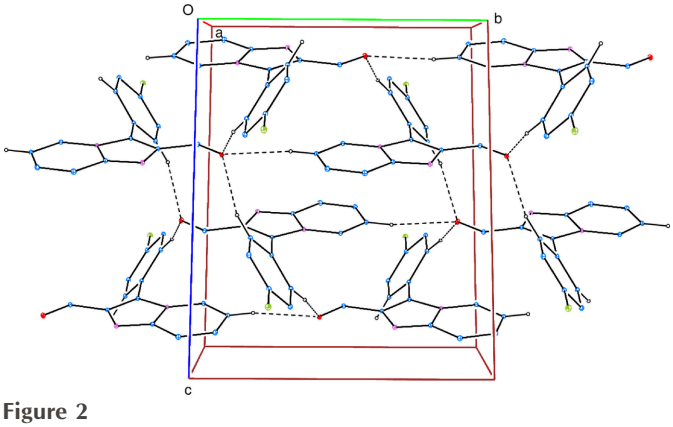


Figure 2
A partial packing diagram of (**I**) viewed down the *a*-axis direction with C—H···O hydrogen bonds shown as dashed lines. Hydrogen atoms not involved in these interactions have been omitted for clarity.

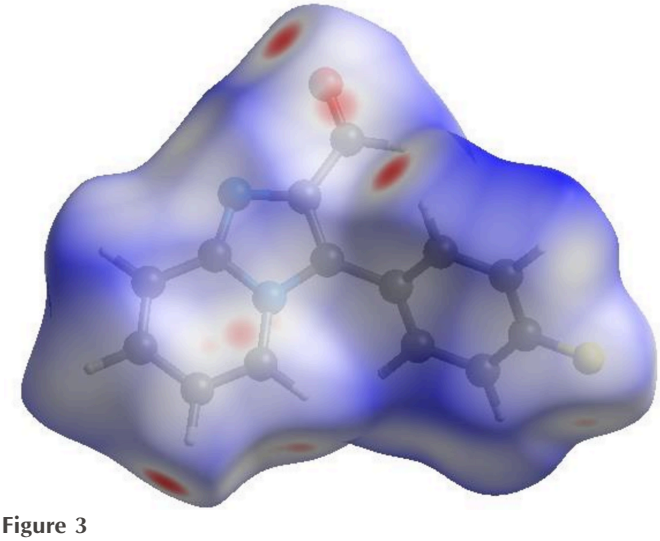


Figure 3
View of the three-dimensional Hirshfeld surface of (**I**) plotted over d_{norm}.

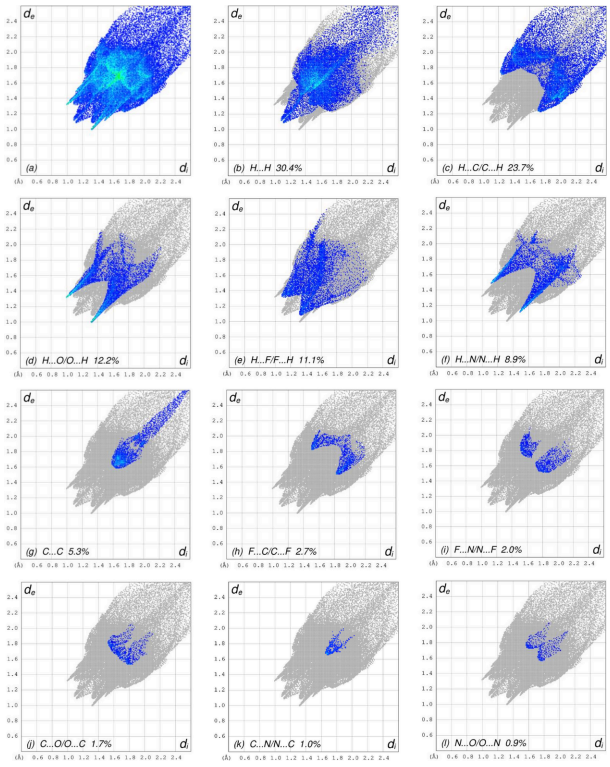


Figure 4
The two-dimensional fingerprint plots for (**I**), showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) H···C/C···H, (*d*) H···O/O···H, (*e*) H···F/F···H, (*f*) H···N/N···H, (*g*) C···C, (*h*) F···C/C···F, (*i*) F···N/N···F, (*j*) C···O/O···C, (*k*) C···N/N···C and (*l*) N···O/O···N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_9FN_2O$
M_r	240.23
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	8.97833 (9), 10.13609 (9), 12.85096 (15)
β (°)	110.3575 (13)
V (Å^3)	1096.46 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.88
Crystal size (mm)	0.39 × 0.30 × 0.16
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.153, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13951, 2296, 2221
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.096, 1.07
No. of reflections	2296
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.17

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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full crystallographic data

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3-(4-Fluorophenyl)imidazo[1,2-*a*]pyridine-2-carbaldehyde

Crystal data

$C_{14}H_9FN_2O$
M_r = 240.23
Monoclinic, $P2_1/n$
a = 8.97833 (9) Å
b = 10.13609 (9) Å
c = 12.85096 (15) Å
β = 110.3575 (13)°
V = 1096.46 (2) Å^3
Z = 4
$F(000)$ = 496
D_x = 1.455 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 10360 reflections
θ = 5.2–76.8°
μ = 0.88 mm^{-1}
T = 100 K
Prism, colorless
0.39 × 0.30 × 0.16 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2023)
T_{min} = 0.153, T_{max} = 1.000
13951 measured reflections
2296 independent reflections
2221 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 77.0°, θ_{min} = 5.3°
h = −10→11
k = −12→12
l = −16→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.07
2296 reflections
164 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.4295P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL2018/3* (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0041 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The C-bound hydrogen atom positions were calculated geometrically (C—H = 0.95 Å) and refined using a riding model by applying the constraint of $U_{iso}(H) = 1.2U_{eq}(C)$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
F14	0.94015 (9)	0.74538 (7)	0.18719 (7)	0.0262 (2)
O10	0.32012 (9)	1.07889 (8)	0.39594 (7)	0.0216 (2)
N4	0.37034 (10)	0.63485 (9)	0.37344 (7)	0.0149 (2)
N1	0.24663 (11)	0.80627 (9)	0.42026 (8)	0.0175 (2)
C3	0.44194 (13)	0.74722 (10)	0.35264 (9)	0.0156 (2)
C2	0.36309 (12)	0.85016 (11)	0.38211 (8)	0.0157 (2)
C11	0.57305 (13)	0.74542 (10)	0.30875 (10)	0.0165 (2)
C8A	0.25290 (12)	0.67653 (11)	0.41485 (9)	0.0169 (2)
C12	0.55893 (13)	0.67604 (11)	0.21145 (9)	0.0188 (2)
H12	0.464190	0.628630	0.173733	0.023*
C5	0.40337 (12)	0.50308 (10)	0.36430 (9)	0.0167 (2)
H5	0.485900	0.478047	0.337956	0.020*
C15	0.83753 (13)	0.81605 (11)	0.32299 (10)	0.0209 (3)
H15	0.932676	0.863295	0.359970	0.025*
C6	0.31690 (13)	0.41015 (11)	0.39326 (9)	0.0186 (2)
H6	0.338214	0.319273	0.386936	0.022*
C16	0.71280 (13)	0.81545 (10)	0.36391 (9)	0.0186 (2)
H16	0.722589	0.862899	0.429719	0.022*
C13	0.68258 (14)	0.67605 (11)	0.16965 (9)	0.0205 (2)
H13	0.673737	0.629443	0.103622	0.025*
C14	0.81886 (14)	0.74598 (10)	0.22711 (10)	0.0198 (3)
C9	0.39053 (12)	0.99003 (11)	0.36905 (9)	0.0171 (2)
H9	0.468252	1.013253	0.337566	0.021*
C8	0.16225 (13)	0.57789 (12)	0.44436 (10)	0.0219 (3)
H8	0.080367	0.602209	0.471573	0.026*
C7	0.19366 (14)	0.44852 (12)	0.43332 (10)	0.0222 (3)
H7	0.132621	0.382589	0.452579	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F14	0.0238 (4)	0.0262 (4)	0.0375 (4)	−0.0010 (3)	0.0220 (3)	−0.0008 (3)
O10	0.0223 (4)	0.0171 (4)	0.0256 (4)	0.0027 (3)	0.0084 (3)	−0.0022 (3)
N4	0.0147 (4)	0.0141 (5)	0.0167 (4)	−0.0005 (3)	0.0068 (3)	0.0006 (3)
N1	0.0165 (4)	0.0179 (5)	0.0199 (4)	0.0003 (3)	0.0089 (4)	−0.0002 (3)
C3	0.0153 (5)	0.0149 (5)	0.0167 (5)	−0.0009 (4)	0.0060 (4)	0.0006 (4)
C2	0.0147 (5)	0.0167 (5)	0.0161 (5)	0.0005 (4)	0.0060 (4)	−0.0003 (4)

C11	0.0165 (5)	0.0143 (5)	0.0211 (5)	0.0022 (4)	0.0094 (4)	0.0030 (4)
C8A	0.0153 (5)	0.0194 (5)	0.0177 (5)	0.0007 (4)	0.0080 (4)	0.0001 (4)
C12	0.0191 (5)	0.0167 (5)	0.0224 (5)	−0.0007 (4)	0.0095 (4)	0.0003 (4)
C5	0.0174 (5)	0.0151 (5)	0.0180 (5)	0.0017 (4)	0.0066 (4)	−0.0005 (4)
C15	0.0177 (5)	0.0172 (5)	0.0296 (6)	−0.0009 (4)	0.0104 (4)	0.0004 (4)
C6	0.0210 (5)	0.0149 (5)	0.0189 (5)	−0.0001 (4)	0.0057 (4)	0.0002 (4)
C16	0.0191 (5)	0.0154 (5)	0.0232 (5)	0.0002 (4)	0.0096 (4)	−0.0004 (4)
C13	0.0249 (6)	0.0171 (5)	0.0237 (5)	0.0004 (4)	0.0138 (5)	−0.0003 (4)
C14	0.0192 (5)	0.0172 (5)	0.0292 (6)	0.0026 (4)	0.0162 (5)	0.0043 (4)
C9	0.0163 (5)	0.0167 (5)	0.0184 (5)	0.0004 (4)	0.0061 (4)	−0.0003 (4)
C8	0.0204 (5)	0.0232 (6)	0.0269 (6)	−0.0012 (4)	0.0142 (5)	0.0009 (4)
C7	0.0226 (5)	0.0210 (6)	0.0255 (6)	−0.0045 (4)	0.0116 (4)	0.0020 (4)

Geometric parameters (Å, º)

F14—C14	1.3556 (13)	C5—H5	0.9500
O10—C9	1.2175 (14)	C5—C6	1.3527 (15)
N4—C3	1.3790 (13)	C15—H15	0.9500
N4—C8A	1.4013 (13)	C15—C16	1.3930 (15)
N4—C5	1.3820 (13)	C15—C14	1.3810 (17)
N1—C2	1.3741 (13)	C6—H6	0.9500
N1—C8A	1.3192 (15)	C6—C7	1.4272 (15)
C3—C2	1.3855 (15)	C16—H16	0.9500
C3—C11	1.4718 (14)	C13—H13	0.9500
C2—C9	1.4587 (15)	C13—C14	1.3843 (17)
C11—C12	1.4014 (15)	C9—H9	0.9500
C11—C16	1.4006 (15)	C8—H8	0.9500
C8A—C8	1.4215 (15)	C8—C7	1.3591 (17)
C12—H12	0.9500	C7—H7	0.9500
C12—C13	1.3914 (15)		
C3—N4—C8A	106.72 (9)	C14—C15—H15	121.0
C3—N4—C5	130.80 (9)	C14—C15—C16	118.08 (10)
C5—N4—C8A	122.39 (9)	C5—C6—H6	120.0
C8A—N1—C2	104.61 (9)	C5—C6—C7	120.05 (10)
N4—C3—C2	104.59 (9)	C7—C6—H6	120.0
N4—C3—C11	123.57 (9)	C11—C16—H16	119.8
C2—C3—C11	131.83 (9)	C15—C16—C11	120.49 (10)
N1—C2—C3	112.23 (10)	C15—C16—H16	119.8
N1—C2—C9	122.39 (10)	C12—C13—H13	121.0
C3—C2—C9	125.28 (10)	C14—C13—C12	118.03 (10)
C12—C11—C3	120.80 (10)	C14—C13—H13	121.0
C16—C11—C3	119.66 (10)	F14—C14—C15	118.41 (10)
C16—C11—C12	119.54 (10)	F14—C14—C13	118.24 (10)
N4—C8A—C8	117.75 (10)	C15—C14—C13	123.35 (10)
N1—C8A—N4	111.84 (9)	O10—C9—C2	124.14 (10)
N1—C8A—C8	130.41 (10)	O10—C9—H9	117.9
C11—C12—H12	119.7	C2—C9—H9	117.9

C13—C12—C11	120.52 (10)	C8A—C8—H8	120.3
C13—C12—H12	119.7	C7—C8—C8A	119.47 (10)
N4—C5—H5	120.4	C7—C8—H8	120.3
C6—C5—N4	119.27 (10)	C6—C7—H7	119.5
C6—C5—H5	120.4	C8—C7—C6	121.04 (10)
C16—C15—H15	121.0	C8—C7—H7	119.5

N4—C3—C2—N1	0.12 (12)	C11—C12—C13—C14	0.17 (16)
N4—C3—C2—C9	−176.34 (9)	C8A—N4—C3—C2	−0.30 (11)
N4—C3—C11—C12	53.26 (15)	C8A—N4—C3—C11	179.59 (10)
N4—C3—C11—C16	−127.70 (11)	C8A—N4—C5—C6	1.88 (16)
N4—C8A—C8—C7	1.04 (16)	C8A—N1—C2—C3	0.12 (12)
N4—C5—C6—C7	−0.33 (16)	C8A—N1—C2—C9	176.70 (10)
N1—C2—C9—O10	4.15 (17)	C8A—C8—C7—C6	0.41 (17)
N1—C8A—C8—C7	−178.51 (11)	C12—C11—C16—C15	−0.27 (16)
C3—N4—C8A—N1	0.41 (12)	C12—C13—C14—F14	179.47 (9)
C3—N4—C8A—C8	−179.23 (10)	C12—C13—C14—C15	−0.43 (17)
C3—N4—C5—C6	178.09 (10)	C5—N4—C3—C2	−176.96 (10)
C3—C2—C9—O10	−179.73 (10)	C5—N4—C3—C11	2.94 (18)
C3—C11—C12—C13	179.21 (10)	C5—N4—C8A—N1	177.41 (9)
C3—C11—C16—C15	−179.32 (10)	C5—N4—C8A—C8	−2.23 (15)
C2—N1—C8A—N4	−0.32 (11)	C5—C6—C7—C8	−0.80 (17)
C2—N1—C8A—C8	179.26 (11)	C16—C11—C12—C13	0.17 (16)
C2—C3—C11—C12	−126.88 (13)	C16—C15—C14—F14	−179.57 (9)
C2—C3—C11—C16	52.16 (17)	C16—C15—C14—C13	0.33 (17)
C11—C3—C2—N1	−179.76 (11)	C14—C15—C16—C11	0.03 (16)
C11—C3—C2—C9	3.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O10^{i}	0.95	2.45	3.3579 (14)	160
C12—H12···O10^{ii}	0.95	2.45	3.3449 (15)	158
C16—H16···O10^{iii}	0.95	2.47	3.3754 (14)	159
C5—H5···F14^{iv}	0.95	2.50	3.1435 (13)	125
C9—H9···F14^{v}	0.95	2.55	3.2089 (14)	127

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) −*x*+3/2, *y*−1/2, −*z*+1/2; (v) −*x*+3/2, *y*+1/2, −*z*+1/2.