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1-Acetyl-3-[2-(2,3,5,6-tetrafluorophenyl)hydrazin-1-ylidene]indolin-2-one

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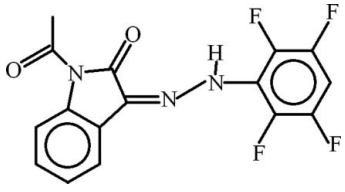
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; R factor = 0.046; wR factor = 0.080; data-to-parameter ratio = 6.4.

In the title compound, $C_{16}H_9F_4N_3O_2$, the dihedral angle between the aromatic ring systems is 4.10 (14)° and a bifurcated intramolecular N—H···(O,F) hydrogen bond generates an S(6) ring for the O-atom acceptor and an S(5) ring for the F-atom acceptor. A short C—H···O conact also occurs. In the crystal, molecules are linked by C—H···O interactions.

Related literature

For background on related isatin derivatives, see: Pervez *et al.* (2007, 2008, 2010*a*). For related structures, see: Abad *et al.* (2006); Pervez *et al.* (2010*b*). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_9F_4N_3O_2$
M_r = 351.26
Monoclinic, $P2_1$
a = 9.8993 (19) Å
b = 4.7740 (6) Å
c = 16.066 (3) Å
β = 104.807 (8)°
V = 734.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 296 K
0.32 × 0.24 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.942, T_{max} = 0.952
6095 measured reflections
1462 independent reflections
749 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.080
S = 0.96
1462 reflections
227 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.86	1.99	2.694 (5)	139
N2—H2···F1	0.86	2.29	2.658 (5)	106
C6—H6···O1	0.93	2.33	2.857 (8)	116
C14—H14···O1^{i}	0.93	2.32	3.217 (7)	163

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5498).

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supporting information

Acta Cryst. (2010). E**66**, o1686 [doi:10.1107/S1600536810022580]

1-Acetyl-3-[2-(2,3,5,6-tetrafluorophenyl)hydrazin-1-ylidene]indolin-2-one

Humayun Pervez, Muhammad Yaqub, Maqbool Ahmad, M. Nawaz Tahir and Robina Akhtar

S1. Comment

In continuation of our previous work on the synthesis of isatin derivatives having physiological properties (Pervez *et al.*, 2007, 2008, 2010*a*, 2010*b*), we report herein the synthesis and crystal structure of the title compound (I), (Fig. 1).

The crystal structure of *N*-(2-chloropyrid-4-yl)-*N*′-(2,3,5,6-tetrafluorophenyl)urea (Abad *et al.*, 2006) has been published which contains the same flouro substituted phenyl group as in (I).

In (I), the 2-oxoindolin-3-hydrazono group A (N3/C1–C8/O2/N1/N2) and tetrafluorophenyl B (C11—C16/F1–F4) are planar with r. m. s. deviations of 0.0197 and 0.0121 Å, respectively. The dihedral angle between A/B is 4.10 (14)°. The acetyl moiety (O1/C9/C10) is oriented at 6.21 (83)° with its parent group A. One S(5) ring motif (Bernstein *et al.*, 1995) is formed due to intramolecular H-bonding of N—H···F type, two S(6) ring motifs due to N—H···O and C—H···O interactions (Table 1, Fig. 1) are formed. The molecules are stabilized in the form of one dimensional polymeric chains extending along the *a* axis (Fig. 2).

S2. Experimental

A solution of 1-acetylisatin (0.95 g, 5.0 mmol) in ethanol (50 ml) was added to the solution of 2,3,5,6-tetrafluorophenyl hydrazine (0.90 g, 5.0 mmol) made in concentrated sulfuric acid (8 ml) and diluted with ethanol (50 ml). The reaction mixture was then refluxed for 30 min. The bright yellow crystalline solid formed during refluxing was collected by suction filtration. Thorough washing with hot ethanol furnished the desired compound (I) in pure form (0.40 g, 23%), m.p. 445 K. Bright yellow prisms of (I) were grown in chloroform by slow evaporation method at room temperature.

S3. Refinement

In the absence of anamolous scattering, the Friedal pairs were merged before refinement. The H-atoms were positioned geometrically (N–H = 0.86 Å, C–H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl and x = 1.2 for all other H-atoms.

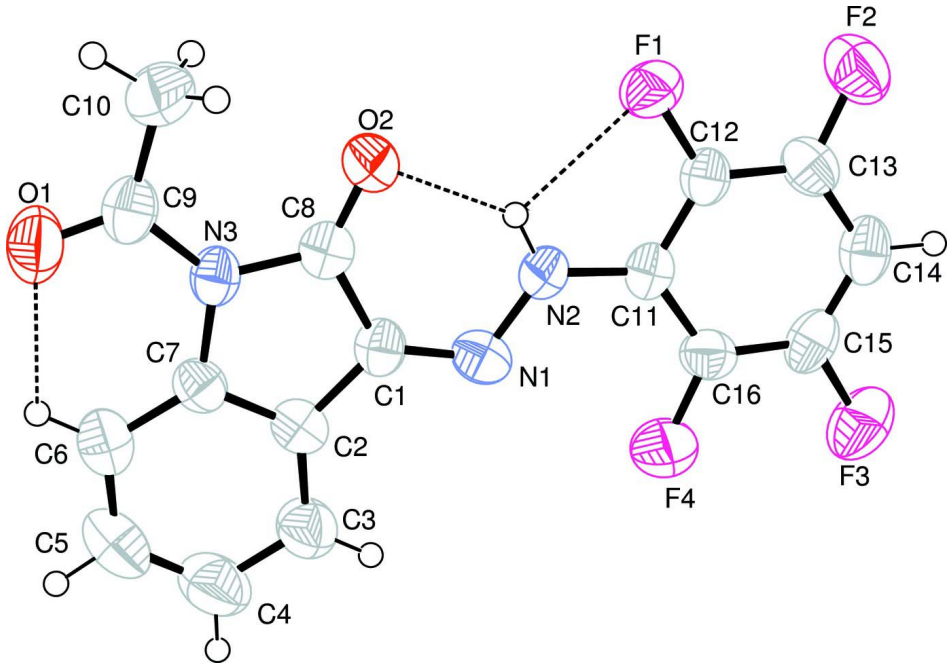

Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level. The dotted lines indicate the intra-molecular H-bondings.

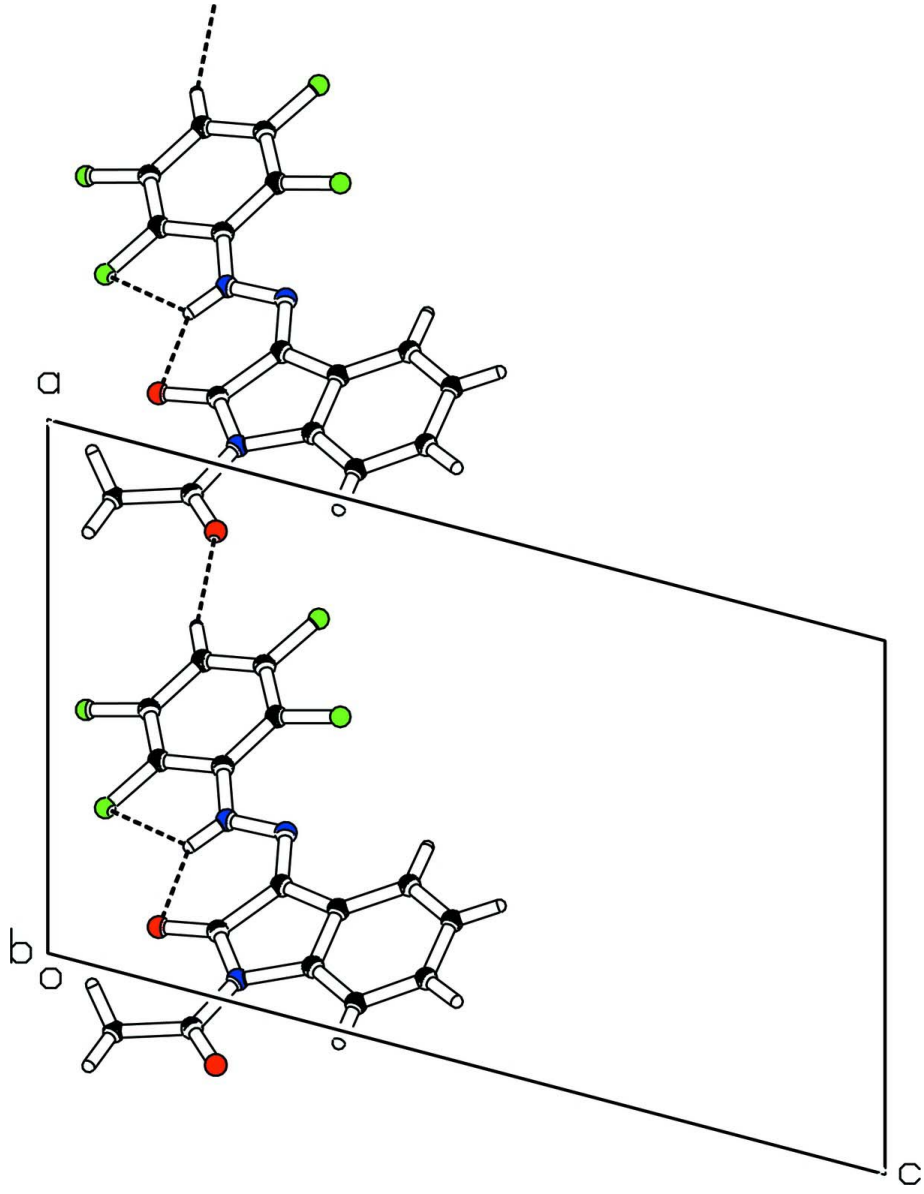

Figure 2

The partial packing of (I), which shows that molecules form one-dimensional polymeric chains extending along the *a* axis.

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$C_{16}H_9F_4N_3O_2$
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Hall symbol: P 2yb
$a = 9.8993$ (19) Å
$b = 4.7740$ (6) Å
$c = 16.066$ (3) Å
$\beta = 104.807$ (8)°
$V = 734.0$ (2) Å^3
$Z = 2$
$F(000) = 356$
$D_x = 1.589$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 749 reflections
$\theta = 2.6$–25.3°
$\mu = 0.14$ mm^{-1}

T = 296 K
Prism, yellow

0.32 × 0.24 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.942, T_{max} = 0.952

6095 measured reflections
1462 independent reflections
749 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 25.3°, θ_{min} = 2.1°
h = −11→11
k = −5→5
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.080
S = 0.96
1462 reflections
227 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-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 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
F1	0.3005 (3)	−0.4142 (6)	0.06872 (17)	0.0638 (12)
F2	0.4748 (3)	−0.8205 (7)	0.0458 (2)	0.0791 (16)
F3	0.7604 (3)	−0.6503 (7)	0.3236 (2)	0.0843 (14)
F4	0.5877 (3)	−0.2576 (8)	0.34942 (19)	0.0820 (14)
O1	−0.1264 (4)	0.8071 (9)	0.2007 (3)	0.0780 (17)
O2	0.1041 (3)	0.1514 (8)	0.1326 (2)	0.0613 (14)
N1	0.3432 (4)	0.0297 (9)	0.2843 (3)	0.0520 (17)
N2	0.3382 (4)	−0.1238 (9)	0.2140 (3)	0.0492 (17)
N3	0.0446 (4)	0.4800 (9)	0.2270 (3)	0.0472 (17)
C1	0.2424 (5)	0.2126 (11)	0.2794 (3)	0.045 (2)
C2	0.2254 (6)	0.3936 (11)	0.3472 (3)	0.048 (2)
C3	0.3062 (6)	0.4269 (12)	0.4321 (4)	0.067 (3)
C4	0.2608 (7)	0.6204 (15)	0.4832 (4)	0.085 (3)
C5	0.1400 (8)	0.7727 (13)	0.4520 (4)	0.082 (3)

C6	0.0581 (6)	0.7417 (12)	0.3682 (4)	0.064 (3)
C7	0.1054 (5)	0.5532 (10)	0.3168 (3)	0.046 (2)
C8	0.1251 (5)	0.2667 (11)	0.2034 (3)	0.047 (2)
C9	−0.0694 (6)	0.6214 (14)	0.1721 (4)	0.057 (2)
C10	−0.1145 (5)	0.5317 (13)	0.0791 (3)	0.078 (3)
C11	0.4358 (5)	−0.3222 (11)	0.2091 (3)	0.0415 (19)
C12	0.4162 (5)	−0.4726 (11)	0.1331 (3)	0.046 (2)
C13	0.5063 (6)	−0.6775 (12)	0.1212 (4)	0.055 (2)
C14	0.6244 (6)	−0.7476 (12)	0.1850 (4)	0.058 (3)
C15	0.6455 (6)	−0.5977 (13)	0.2589 (4)	0.055 (2)
C16	0.5565 (6)	−0.3921 (11)	0.2732 (3)	0.052 (2)
H2	0.26986	−0.09659	0.16945	0.0589*
H3	0.38710	0.32286	0.45329	0.0808*
H4	0.31279	0.64831	0.53964	0.1016*
H5	0.11238	0.90027	0.48823	0.0984*
H6	−0.02410	0.84251	0.34781	0.0768*
H10A	−0.19107	0.64643	0.04892	0.1164*
H10B	−0.14341	0.33917	0.07589	0.1164*
H10C	−0.03772	0.55230	0.05320	0.1164*
H14	0.68534	−0.88833	0.17772	0.0692*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.059 (2)	0.066 (2)	0.058 (2)	0.0084 (17)	−0.0006 (16)	0.0018 (17)
F2	0.096 (3)	0.066 (2)	0.078 (3)	0.010 (2)	0.027 (2)	−0.013 (2)
F3	0.062 (2)	0.101 (3)	0.083 (2)	0.018 (2)	0.006 (2)	0.024 (2)
F4	0.077 (2)	0.098 (3)	0.060 (2)	0.013 (2)	−0.0025 (19)	−0.010 (2)
O1	0.075 (3)	0.076 (3)	0.083 (3)	0.029 (3)	0.020 (2)	0.002 (2)
O2	0.061 (2)	0.070 (3)	0.052 (2)	0.010 (2)	0.013 (2)	−0.010 (2)
N1	0.062 (3)	0.047 (3)	0.048 (3)	−0.006 (3)	0.016 (2)	−0.002 (3)
N2	0.042 (3)	0.055 (3)	0.048 (3)	0.008 (2)	0.007 (2)	−0.001 (3)
N3	0.048 (3)	0.039 (3)	0.055 (3)	0.008 (2)	0.014 (3)	0.006 (2)
C1	0.048 (4)	0.038 (4)	0.051 (4)	0.003 (3)	0.015 (3)	0.008 (3)
C2	0.058 (4)	0.043 (4)	0.045 (4)	−0.004 (3)	0.018 (3)	0.002 (3)
C3	0.075 (4)	0.069 (5)	0.055 (4)	0.010 (4)	0.011 (4)	−0.004 (4)
C4	0.105 (6)	0.083 (6)	0.062 (5)	0.003 (5)	0.014 (4)	−0.015 (4)
C5	0.112 (6)	0.068 (5)	0.073 (5)	0.001 (5)	0.035 (4)	−0.020 (4)
C6	0.067 (4)	0.056 (4)	0.074 (5)	0.007 (3)	0.027 (4)	−0.003 (4)
C7	0.053 (4)	0.037 (4)	0.050 (4)	−0.004 (3)	0.016 (3)	−0.001 (3)
C8	0.049 (4)	0.042 (4)	0.051 (4)	−0.003 (3)	0.017 (3)	0.006 (3)
C9	0.051 (4)	0.057 (4)	0.066 (4)	0.005 (3)	0.022 (3)	0.008 (3)
C10	0.072 (4)	0.095 (5)	0.057 (4)	0.019 (4)	0.000 (3)	0.000 (4)
C11	0.040 (3)	0.037 (3)	0.051 (4)	−0.001 (3)	0.018 (3)	0.010 (3)
C12	0.046 (4)	0.040 (4)	0.052 (4)	0.006 (3)	0.013 (3)	0.006 (3)
C13	0.068 (4)	0.045 (4)	0.055 (4)	0.001 (3)	0.020 (4)	−0.002 (3)
C14	0.057 (4)	0.044 (4)	0.078 (5)	0.008 (3)	0.028 (4)	0.012 (4)
C15	0.044 (4)	0.057 (4)	0.064 (4)	0.009 (3)	0.012 (3)	0.021 (4)

C16	0.051 (4)	0.056 (4)	0.048 (4)	−0.009 (3)	0.013 (3)	−0.006 (3)

Geometric parameters (Å, º)

F1—C12	1.362 (6)	C4—C5	1.380 (10)
F2—C13	1.356 (7)	C5—C6	1.391 (9)
F3—C15	1.354 (7)	C6—C7	1.382 (8)
F4—C16	1.347 (6)	C9—C10	1.508 (8)
O1—C9	1.203 (8)	C11—C16	1.404 (7)
O2—C8	1.233 (6)	C11—C12	1.387 (7)
N1—N2	1.337 (6)	C12—C13	1.370 (8)
N1—C1	1.313 (7)	C13—C14	1.385 (9)
N2—C11	1.370 (7)	C14—C15	1.356 (9)
N3—C7	1.457 (7)	C15—C16	1.377 (8)
N3—C8	1.404 (7)	C3—H3	0.9300
N3—C9	1.414 (8)	C4—H4	0.9300
N2—H2	0.8600	C5—H5	0.9300
C1—C8	1.477 (7)	C6—H6	0.9300
C1—C2	1.434 (7)	C10—H10A	0.9600
C2—C7	1.391 (8)	C10—H10B	0.9600
C2—C3	1.403 (8)	C10—H10C	0.9600
C3—C4	1.385 (9)	C14—H14	0.9300
N2—N1—C1	116.8 (4)	F1—C12—C13	119.4 (5)
N1—N2—C11	123.6 (4)	C11—C12—C13	122.9 (5)
C7—N3—C8	108.8 (4)	F1—C12—C11	117.7 (4)
C7—N3—C9	124.4 (4)	C12—C13—C14	121.7 (5)
C8—N3—C9	126.6 (5)	F2—C13—C12	118.4 (5)
N1—N2—H2	118.00	F2—C13—C14	119.9 (5)
C11—N2—H2	118.00	C13—C14—C15	115.6 (6)
N1—C1—C2	126.2 (5)	F3—C15—C16	116.9 (5)
N1—C1—C8	126.2 (5)	C14—C15—C16	124.1 (6)
C2—C1—C8	107.6 (4)	F3—C15—C14	119.1 (5)
C1—C2—C3	131.2 (5)	C11—C16—C15	120.5 (5)
C3—C2—C7	120.2 (5)	F4—C16—C11	120.5 (5)
C1—C2—C7	108.6 (4)	F4—C16—C15	118.9 (5)
C2—C3—C4	117.5 (6)	C2—C3—H3	121.00
C3—C4—C5	121.2 (6)	C4—C3—H3	121.00
C4—C5—C6	122.2 (6)	C3—C4—H4	119.00
C5—C6—C7	116.4 (6)	C5—C4—H4	119.00
C2—C7—C6	122.5 (5)	C4—C5—H5	119.00
N3—C7—C2	108.4 (4)	C6—C5—H5	119.00
N3—C7—C6	129.1 (5)	C5—C6—H6	122.00
N3—C8—C1	106.5 (4)	C7—C6—H6	122.00
O2—C8—N3	126.9 (5)	C9—C10—H10A	110.00
O2—C8—C1	126.6 (5)	C9—C10—H10B	109.00
O1—C9—N3	119.4 (6)	C9—C10—H10C	109.00
O1—C9—C10	122.6 (6)	H10A—C10—H10B	109.00

N3—C9—C10	118.0 (5)	H10A—C10—H10C	109.00
N2—C11—C12	117.8 (4)	H10B—C10—H10C	109.00
C12—C11—C16	115.1 (5)	C13—C14—H14	122.00
N2—C11—C16	127.1 (5)	C15—C14—H14	122.00
C1—N1—N2—C11	179.8 (5)	C1—C2—C7—C6	177.8 (5)
N2—N1—C1—C2	178.5 (5)	C3—C2—C7—N3	178.8 (5)
N2—N1—C1—C8	−0.7 (8)	C3—C2—C7—C6	−2.1 (8)
N1—N2—C11—C12	178.2 (5)	C2—C3—C4—C5	0.6 (10)
N1—N2—C11—C16	−2.2 (8)	C3—C4—C5—C6	−0.3 (11)
C8—N3—C7—C2	2.2 (6)	C4—C5—C6—C7	−1.2 (10)
C8—N3—C7—C6	−176.7 (5)	C5—C6—C7—N3	−178.8 (5)
C9—N3—C7—C2	−172.7 (5)	C5—C6—C7—C2	2.4 (8)
C9—N3—C7—C6	8.4 (8)	N2—C11—C12—F1	−0.5 (7)
C7—N3—C8—O2	179.4 (5)	N2—C11—C12—C13	−179.4 (5)
C7—N3—C8—C1	−2.2 (5)	C16—C11—C12—F1	179.8 (4)
C9—N3—C8—O2	−5.9 (9)	C16—C11—C12—C13	0.9 (8)
C9—N3—C8—C1	172.6 (5)	N2—C11—C16—F4	−0.6 (8)
C7—N3—C9—O1	−3.7 (9)	N2—C11—C16—C15	180.0 (5)
C7—N3—C9—C10	176.2 (5)	C12—C11—C16—F4	179.1 (5)
C8—N3—C9—O1	−177.7 (5)	C12—C11—C16—C15	−0.4 (8)
C8—N3—C9—C10	2.3 (8)	F1—C12—C13—F2	−1.6 (8)
N1—C1—C2—C3	0.5 (10)	F1—C12—C13—C14	−179.1 (5)
N1—C1—C2—C7	−179.4 (5)	C11—C12—C13—F2	177.3 (5)
C8—C1—C2—C3	179.8 (6)	C11—C12—C13—C14	−0.2 (9)
C8—C1—C2—C7	0.0 (6)	F2—C13—C14—C15	−178.4 (5)
N1—C1—C8—O2	−0.8 (9)	C12—C13—C14—C15	−0.9 (9)
N1—C1—C8—N3	−179.3 (5)	C13—C14—C15—F3	−178.6 (5)
C2—C1—C8—O2	179.9 (5)	C13—C14—C15—C16	1.5 (9)
C2—C1—C8—N3	1.4 (6)	F3—C15—C16—F4	−0.2 (8)
C1—C2—C3—C4	−179.3 (6)	F3—C15—C16—C11	179.3 (5)
C7—C2—C3—C4	0.6 (9)	C14—C15—C16—F4	179.7 (5)
C1—C2—C7—N3	−1.3 (6)	C14—C15—C16—C11	−0.9 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	1.99	2.694 (5)	139
N2—H2···F1	0.86	2.29	2.658 (5)	106
C6—H6···O1	0.93	2.33	2.857 (8)	116
C14—H14···O1^{i}	0.93	2.32	3.217 (7)	163

Symmetry code: (i) *x*+1, *y*−2, *z*.