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2-(4-Fluoroanilino)-3-(2-hydroxyethyl)-quinazolin-4(3*H*)-one

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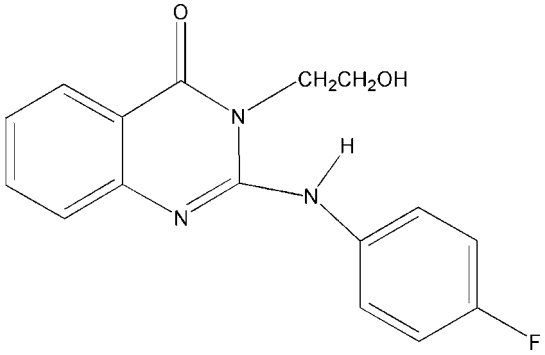
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.102; data-to-parameter ratio = 14.5.

The molecular and crystal structures of the title compound, $C_{16}H_{14}FN_3O_2$, are stabilized by intramolecular N—H···O and intermolecular O—H···O hydrogen bonds. The existence of non-classical intramolecular C—H···N hydrogen bonds provides a dihedral angle between the fluoro-substituted benzene and pyrimidinone rings of 7.9 (1)°.

Related literature

For the pharmacological activity of N3 and C7 disubstituted quinazolines, see: Usha *et al.* (2006). For the synthesis of quinazolinone and thienopyrimidinones, see: Yang *et al.* (2008). For synthesis, drug discovery and crystal structures, see: Yang & Wu (2008); Wang *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{14}FN_3O_2$
M_r = 299.30
Monoclinic, $P2_1/n$
a = 8.5737 (8) Å
b = 10.8268 (10) Å
c = 15.2490 (13) Å
β = 104.070(10)°
V = 1371.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 273 K
0.10 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
8342 measured reflections
2976 independent reflections
2475 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.102
S = 1.06
2976 reflections
205 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···N3	0.93	2.28	2.8807 (16)	122
N1—H1···O2	0.881 (9)	1.982 (10)	2.8253 (14)	159.7 (13)
O2—H2*A*···O1^{i}	0.837 (9)	1.909 (10)	2.7426 (13)	173.6 (18)

Symmetry code: (i) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2120).

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Yang, X. H., Wu, M. H., Sun, S. F., Ding, M. W., Xie, J. L. & Xia, Q. H. (2008). *J. Heterocycl. Chem.* **5**, 1365–1369.

supporting information

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S1. Comment

Quinazolone derivatives have evoked considerable attention in recent years as these are endowed with wide range of pharmaceutical activities. 3*H*–Quinazolin–4–one represents a useful nucleus for preparation of some new sedative/hypnotic and anticonvulsant agents. Of the various quinazolines reported, the N3 and C7 disubstituted quinazolines exhibit interesting pharmacological activities like analgesic, anti–inflammatory, antibacterial and anticonvulsant activities (Usha *et al.*, 2006). In connection with our ongoing heterocyclic synthesis and drug discovery project (Yang & Wu, 2008), we have focused on the synthesis of quinazolinone and thienopyrimidinones (Wang *et al.*, 2008; Yang *et al.*, 2008). Herein, the title compound was synthesied and its molecular (Fig. 1) and crystal structures were determined. An intramolecular N1—H1···O2 and non–classical C5—H5···N3 hydrogen bonds provide the conformation of the molecule - dihedral angle between the fluoro–substituted benzene ring and pyrimidinone rings as 7.9 (1)°. In the crystal structure, intermolecular O2—H2a···O1^{i} hydrogen bonds link of molecules into chains (see Tab. 1 and Fig. 2). Symmetry code: (i) -x+5/2, y-1/2, -z+1/2.

S2. Experimental

To a solution of 2–ethoxycarbonyliminophosphorane (1.27 g, 3.0 mmol) in 10 ml anhydrous *THF*, 4–fluorophenyl isocyanate (0.41 g, 3.0 mmol) was added dropwise at room temperature. The reaction mixture was left unstirred for 6 h at 273–278 K, whereafter the above resulting solution was added dropwise to a solution of ethanolamine (0.18 g, 3 mmol) in 5 ml anhydrous *THF*. The reaction mixture was stirred overnight at room temperature, the solvent was removed under reduced pressure and the residue was recrystallized from $C_2H_4Cl_2$/CH_3OH (1:1/v:v) to give colourless crystals of the title compound. Yield 89%.

S3. Refinement

The C–bonded H atoms were placed in calculated positions with C—H = 0.93 Å for aromatic, C—H = 0.97 Å for methylene and refined in the riding model approximation. The positional parameters of N– and O–bonded H atoms were found from difference Fourier map and refined independently. For all H atoms U_{iso}(H) = 1.2U_{eq}(C, N) and U_{iso}(H) = 1.5U_{eq}(O).

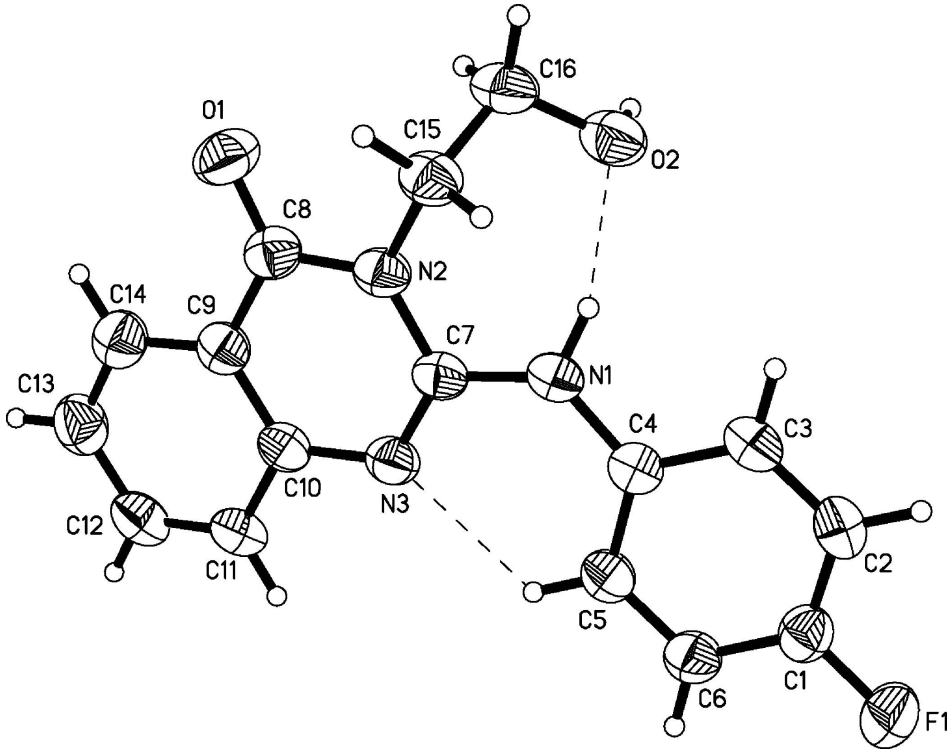

Figure 1

View of the title molecule with the atom–labeling scheme. Dispalcement ellipsoids are drawn at the 50% probability level. H at6oms are presented as a small spheres of arbitrary radius. Intramolecular hydrogen bonds are drawn by dashed lines.

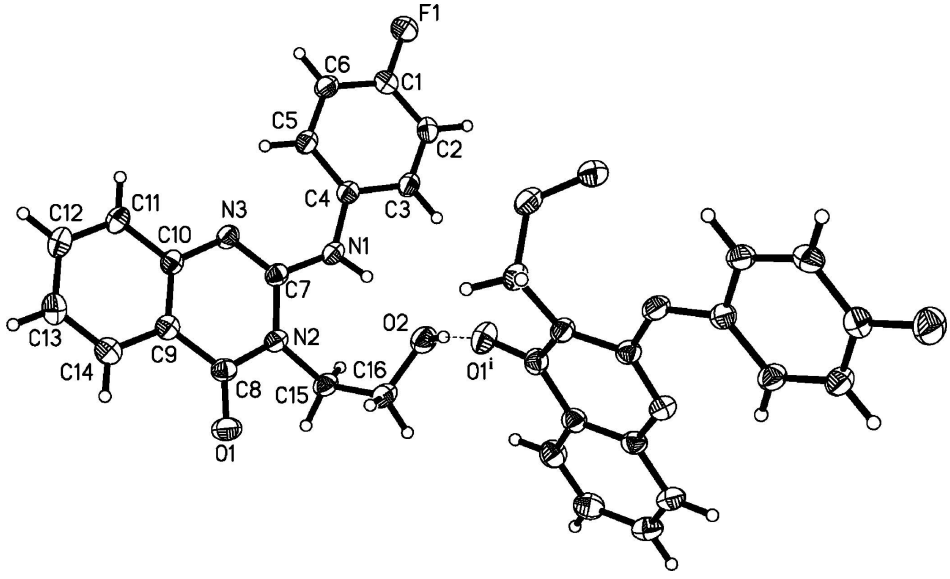

Figure 2

Part of the crystal structure of the title compound showing intermolecular hydrogen bonds as dashed lines. Symmetry code: (i) -x+5/2, y-1/2, -z+1/2.

2-(4-Fluoroanilino)-3-(2-hydroxyethyl)quinazolin-4(3*H*)-one

Crystal data

$C_{16}H_{14}FN_3O_2$
$M_r = 299.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.5737\ (8)$ Å
$b = 10.8268\ (10)$ Å
$c = 15.2490\ (13)$ Å
$\beta = 104.407\ (1)°$
$V = 1371.0\ (2)$ Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.450$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3320 reflections
$\theta = 2.3–29.2°$
$\mu = 0.11$ mm^{-1}
$T = 273$ K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: Fine–focus sealed tube
Graphite monochromator
φ and ω scans
8342 measured reflections
2976 independent reflections
2475 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
$h = -10\rightarrow10$
$k = -12\rightarrow13$
$l = -19\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: Full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.102$
$S = 1.06$
2976 reflections
205 parameters
2 restraints
Primary atom site location: Direct
Secondary atom site location: Difmap
Hydrogen site location: Geom
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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	0.56770 (14)	0.34529 (11)	−0.09177 (9)	0.0442 (3)
C2	0.59291 (15)	0.34162 (11)	0.00070 (9)	0.0455 (3)
H2	0.5444	0.2813	0.0283	0.055*
C3	0.69136 (15)	0.42908 (11)	0.05135 (8)	0.0427 (3)
H3	0.7096	0.4276	0.1141	0.051*
C4	0.76459 (13)	0.52003 (10)	0.01077 (8)	0.0379 (3)

C5	0.73709 (15)	0.52084 (12)	−0.08302 (8)	0.0438 (3)
H5	0.7852	0.5807	−0.1113	0.053*
C6	0.63794 (15)	0.43247 (12)	−0.13419 (8)	0.0458 (3)
H6	0.6193	0.4325	−0.1969	0.055*
C7	0.94732 (13)	0.70237 (10)	0.05017 (7)	0.0377 (3)
C8	1.13407 (14)	0.86392 (11)	0.11798 (8)	0.0418 (3)
C9	1.15360 (14)	0.88561 (11)	0.02741 (8)	0.0402 (3)
C10	1.06202 (13)	0.81559 (11)	−0.04382 (8)	0.0389 (3)
C11	1.08192 (15)	0.83508 (12)	−0.13148 (8)	0.0462 (3)
H11	1.0220	0.7892	−0.1798	0.055*
C12	1.18945 (16)	0.92159 (13)	−0.14591 (9)	0.0520 (3)
H12	1.2025	0.9334	−0.2041	0.062*
C13	1.27943 (17)	0.99209 (13)	−0.07485 (10)	0.0549 (3)
H13	1.3514	1.0510	−0.0857	0.066*
C14	1.26168 (16)	0.97446 (12)	0.01099 (10)	0.0504 (3)
H14	1.3215	1.0216	0.0586	0.060*
C15	0.97517 (16)	0.76912 (12)	0.21207 (8)	0.0450 (3)
H15A	0.8617	0.7484	0.1999	0.054*
H15B	0.9889	0.8505	0.2395	0.054*
C16	1.06872 (18)	0.67775 (13)	0.27916 (8)	0.0526 (3)
H16A	1.1828	0.6861	0.2827	0.063*
H16B	1.0521	0.6944	0.3387	0.063*
F1	0.46983 (11)	0.25850 (8)	−0.14287 (6)	0.0651 (3)
N1	0.85952 (13)	0.60633 (10)	0.07009 (7)	0.0439 (2)
H1	0.8863 (16)	0.5818 (12)	0.1269 (6)	0.053*
N2	1.02153 (12)	0.77481 (9)	0.12481 (6)	0.0393 (2)
N3	0.96041 (12)	0.72240 (9)	−0.03130 (6)	0.0411 (2)
O1	1.20713 (12)	0.91951 (9)	0.18653 (6)	0.0565 (3)
O2	1.01772 (12)	0.55593 (9)	0.25230 (6)	0.0546 (3)
H2A	1.0973 (16)	0.5093 (14)	0.2698 (12)	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0436 (6)	0.0404 (6)	0.0481 (7)	−0.0004 (5)	0.0105 (5)	−0.0022 (5)
C2	0.0492 (7)	0.0396 (6)	0.0504 (7)	0.0018 (5)	0.0174 (6)	0.0082 (5)
C3	0.0499 (7)	0.0442 (6)	0.0356 (6)	0.0072 (5)	0.0135 (5)	0.0067 (5)
C4	0.0379 (6)	0.0404 (6)	0.0360 (6)	0.0046 (5)	0.0104 (5)	0.0012 (5)
C5	0.0492 (7)	0.0480 (7)	0.0357 (6)	−0.0038 (5)	0.0132 (5)	0.0024 (5)
C6	0.0508 (7)	0.0519 (7)	0.0352 (6)	−0.0028 (6)	0.0114 (5)	−0.0017 (5)
C7	0.0370 (6)	0.0411 (6)	0.0333 (6)	0.0052 (5)	0.0058 (4)	0.0015 (5)
C8	0.0416 (6)	0.0426 (6)	0.0409 (6)	0.0040 (5)	0.0094 (5)	−0.0044 (5)
C9	0.0388 (6)	0.0399 (6)	0.0420 (6)	0.0059 (5)	0.0102 (5)	0.0020 (5)
C10	0.0377 (6)	0.0406 (6)	0.0376 (6)	0.0065 (5)	0.0080 (5)	0.0053 (5)
C11	0.0476 (7)	0.0538 (7)	0.0351 (6)	0.0046 (6)	0.0060 (5)	0.0081 (5)
C12	0.0531 (7)	0.0602 (8)	0.0442 (7)	0.0058 (6)	0.0149 (6)	0.0147 (6)
C13	0.0513 (8)	0.0542 (8)	0.0607 (9)	−0.0035 (6)	0.0168 (6)	0.0112 (7)
C14	0.0489 (7)	0.0483 (7)	0.0536 (8)	−0.0022 (6)	0.0121 (6)	−0.0010 (6)

C15	0.0535 (7)	0.0494 (7)	0.0348 (6)	0.0053 (6)	0.0160 (5)	−0.0031 (5)
C16	0.0621 (8)	0.0616 (8)	0.0327 (6)	0.0034 (6)	0.0092 (6)	−0.0009 (6)
F1	0.0736 (6)	0.0599 (5)	0.0592 (5)	−0.0229 (4)	0.0116 (4)	−0.0068 (4)
N1	0.0521 (6)	0.0494 (6)	0.0297 (5)	−0.0036 (5)	0.0092 (4)	0.0026 (4)
N2	0.0434 (5)	0.0436 (5)	0.0316 (5)	0.0037 (4)	0.0104 (4)	−0.0013 (4)
N3	0.0436 (5)	0.0456 (6)	0.0331 (5)	0.0000 (4)	0.0077 (4)	0.0028 (4)
O1	0.0601 (6)	0.0639 (6)	0.0450 (5)	−0.0115 (5)	0.0123 (4)	−0.0152 (4)
O2	0.0629 (6)	0.0552 (6)	0.0421 (5)	0.0091 (4)	0.0062 (4)	0.0055 (4)

Geometric parameters (Å, º)

C1—C6	1.3659 (17)	C9—C14	1.4007 (17)
C1—F1	1.3670 (14)	C10—N3	1.3771 (15)
C1—C2	1.3729 (18)	C10—C11	1.4050 (16)
C2—C3	1.3716 (18)	C11—C12	1.3699 (19)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.3929 (16)	C12—C13	1.391 (2)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.3904 (16)	C13—C14	1.3683 (19)
C4—N1	1.4098 (15)	C13—H13	0.9300
C5—C6	1.3846 (17)	C14—H14	0.9300
C5—H5	0.9300	C15—N2	1.4817 (14)
C6—H6	0.9300	C15—C16	1.5035 (18)
C7—N3	1.2932 (14)	C15—H15A	0.9700
C7—N1	1.3616 (15)	C15—H15B	0.9700
C7—N2	1.3983 (15)	C16—O2	1.4174 (17)
C8—O1	1.2335 (14)	C16—H16A	0.9700
C8—N2	1.3866 (15)	C16—H16B	0.9700
C8—C9	1.4511 (16)	N1—H1	0.881 (9)
C9—C10	1.3952 (16)	O2—H2A	0.837 (9)
C6—C1—F1	119.04 (11)	C12—C11—H11	120.0
C6—C1—C2	122.08 (12)	C10—C11—H11	120.0
F1—C1—C2	118.88 (11)	C11—C12—C13	120.99 (12)
C3—C2—C1	118.42 (11)	C11—C12—H12	119.5
C3—C2—H2	120.8	C13—C12—H12	119.5
C1—C2—H2	120.8	C14—C13—C12	119.76 (12)
C2—C3—C4	121.34 (11)	C14—C13—H13	120.1
C2—C3—H3	119.3	C12—C13—H13	120.1
C4—C3—H3	119.3	C13—C14—C9	120.19 (13)
C5—C4—C3	118.79 (11)	C13—C14—H14	119.9
C5—C4—N1	125.37 (11)	C9—C14—H14	119.9
C3—C4—N1	115.82 (10)	N2—C15—C16	114.93 (10)
C6—C5—C4	119.90 (11)	N2—C15—H15A	108.5
C6—C5—H5	120.0	C16—C15—H15A	108.5
C4—C5—H5	120.0	N2—C15—H15B	108.5
C1—C6—C5	119.46 (11)	C16—C15—H15B	108.5
C1—C6—H6	120.3	H15A—C15—H15B	107.5

C5—C6—H6	120.3	O2—C16—C15	109.98 (11)
N3—C7—N1	121.83 (11)	O2—C16—H16A	109.7
N3—C7—N2	123.86 (10)	C15—C16—H16A	109.7
N1—C7—N2	114.31 (10)	O2—C16—H16B	109.7
O1—C8—N2	119.47 (11)	C15—C16—H16B	109.7
O1—C8—C9	124.96 (11)	H16A—C16—H16B	108.2
N2—C8—C9	115.56 (10)	C7—N1—C4	128.75 (10)
C10—C9—C14	120.21 (11)	C7—N1—H1	115.3 (9)
C10—C9—C8	118.50 (11)	C4—N1—H1	113.5 (9)
C14—C9—C8	121.29 (11)	C8—N2—C7	120.89 (10)
N3—C10—C9	122.90 (10)	C8—N2—C15	116.33 (10)
N3—C10—C11	118.21 (11)	C7—N2—C15	122.56 (10)
C9—C10—C11	118.75 (11)	C7—N3—C10	117.72 (10)
C12—C11—C10	120.09 (12)	C16—O2—H2A	107.5 (13)

C6—C1—C2—C3	0.31 (19)	C12—C13—C14—C9	0.1 (2)
F1—C1—C2—C3	179.97 (10)	C10—C9—C14—C13	−0.76 (19)
C1—C2—C3—C4	0.08 (18)	C8—C9—C14—C13	179.16 (12)
C2—C3—C4—C5	−0.34 (18)	N2—C15—C16—O2	−74.18 (14)
C2—C3—C4—N1	178.41 (11)	N3—C7—N1—C4	−3.84 (19)
C3—C4—C5—C6	0.23 (18)	N2—C7—N1—C4	176.74 (11)
N1—C4—C5—C6	−178.39 (11)	C5—C4—N1—C7	−3.0 (2)
F1—C1—C6—C5	179.92 (11)	C3—C4—N1—C7	178.32 (11)
C2—C1—C6—C5	−0.42 (19)	O1—C8—N2—C7	−174.16 (11)
C4—C5—C6—C1	0.14 (19)	C9—C8—N2—C7	6.98 (16)
O1—C8—C9—C10	−179.80 (11)	O1—C8—N2—C15	11.04 (16)
N2—C8—C9—C10	−1.02 (16)	C9—C8—N2—C15	−167.81 (10)
O1—C8—C9—C14	0.28 (19)	N3—C7—N2—C8	−9.29 (17)
N2—C8—C9—C14	179.06 (11)	N1—C7—N2—C8	170.11 (10)
C14—C9—C10—N3	176.35 (11)	N3—C7—N2—C15	165.17 (11)
C8—C9—C10—N3	−3.57 (17)	N1—C7—N2—C15	−15.43 (15)
C14—C9—C10—C11	0.73 (17)	C16—C15—N2—C8	−94.13 (13)
C8—C9—C10—C11	−179.20 (10)	C16—C15—N2—C7	91.16 (14)
N3—C10—C11—C12	−175.91 (11)	N1—C7—N3—C10	−174.89 (10)
C9—C10—C11—C12	−0.08 (18)	N2—C7—N3—C10	4.47 (17)
C10—C11—C12—C13	−0.6 (2)	C9—C10—N3—C7	1.96 (17)
C11—C12—C13—C14	0.5 (2)	C11—C10—N3—C7	177.61 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N3	0.93	2.28	2.8807 (16)	122
N1—H1···O2	0.88 (1)	1.98 (1)	2.8253 (14)	160 (1)
O2—H2*A*···O1^{i}	0.84 (1)	1.91 (1)	2.7426 (13)	174 (2)

Symmetry code: (i) −*x*+5/2, *y*−1/2, −*z*+1/2.