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2-(3-Nitrophenoxy)quinoxaline

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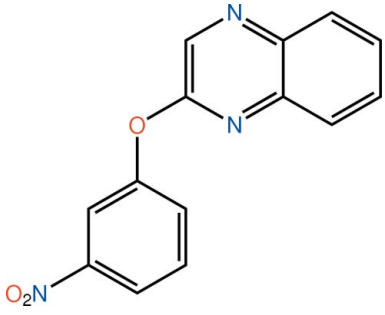
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 8.3.

In the title molecule, $C_{14}H_9N_3O_3$, the dihedral angle between the quinoxaline and benzene rings is 77.13 (9)°. The molecule is twisted about the ether–benzene O—C bond, with a C—O—C—C torsion angle of −102.8 (2)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming layers in the *ab* plane, with one nitro O atom accepting two such interactions. The layers stack along the *c*-axis direction *via* weak C—H···π interactions.

Related literature

For background to the fluorescence properties of compounds related to the title compound, see: Kawai *et al.* (2001); Abdullah (2005). For the structures of the polymorphic phenyl quinoxalin-2-yl ether compound, see: Hassan *et al.* (2008); Abdullah & Ng (2008).



Experimental

Crystal data

$C_{14}H_9N_3O_3$ M_r = 267.24
Monoclinic, $P2_1$
a = 6.0643 (6) Å
b = 5.3676 (5) Å
c = 18.2443 (17) Å
β = 91.780 (1)°
V = 593.58 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
5637 measured reflections
1502 independent reflections
1403 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.04
1501 reflections
181 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*1 is the centroid of the C3–C8 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O2^{i}	0.95	2.34	3.282 (3)	173
C12—H12···O2^{ii}	0.95	2.44	3.159 (2)	133
C5—H5···*Cg*1^{iii}	0.95	2.99	3.696 (2)	133

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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 *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5615).

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S1. Comment

Quinoxaline derivatives show interesting fluorescence properties (Kawai *et al.* 2001; Abdullah, 2005) and this observation prompted the synthesis and characterization of the title compound, (I).

The molecule in (I), Fig. 1, is bent as the quinoxaline ring [r.m.s. deviation = 0.025 Å] forms a dihedral angle of 77.13 (9) ° with the benzene molecule. The twist in the molecule is seen in the value of the C1–O1–C9–C14 torsion angle of -102.8 (2) °. Overall the conformation of the molecule matches those found in the polymorphic phenyl quinoxalin-2-yl ether compound (Hassan *et al.*, 2008; Abdullah & Ng, 2008). In (I), the nitro group is slightly twisted out of the plane of the benzene ring to which it is bonded as seen in the O2–N3–C13–C12 torsion angle of 12.6 (3) °.

The bifurcated nitro-O2 atom is pivotal in the crystal packing, forming two close C–H···O interactions, Table 1, leading to the formation of layers in the *ab* plane, Fig. 2. These stack along the *c* axis, being connected by C–H···π interactions, Fig. 3.

S2. Experimental

3-Nitrophenol (5 mmol) was dissolved in tetrahydrofuran (100 ml) to which was added 2-chloroquinoxaline with a stoichiometric amount of NaOH. The solution was refluxed for 4 h. The mixture was extracted using 5% sodium hydroxide solution (5 ml), then chloroform (20 ml), washed with distilled water (30 ml), and dried over anhydrous sodium hydroxide. Evaporation of the solvent gave a red solid and recrystallization was from its ethanol solution to yield red prisms of (I).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{equiv}(C). In the absence of significant anomalous scattering effects, 1199 Friedel pairs were averaged in the final refinement. In the final refinement a low angle reflection evidently effected by the beam stop were omitted, *i.e.* 0 0 1.

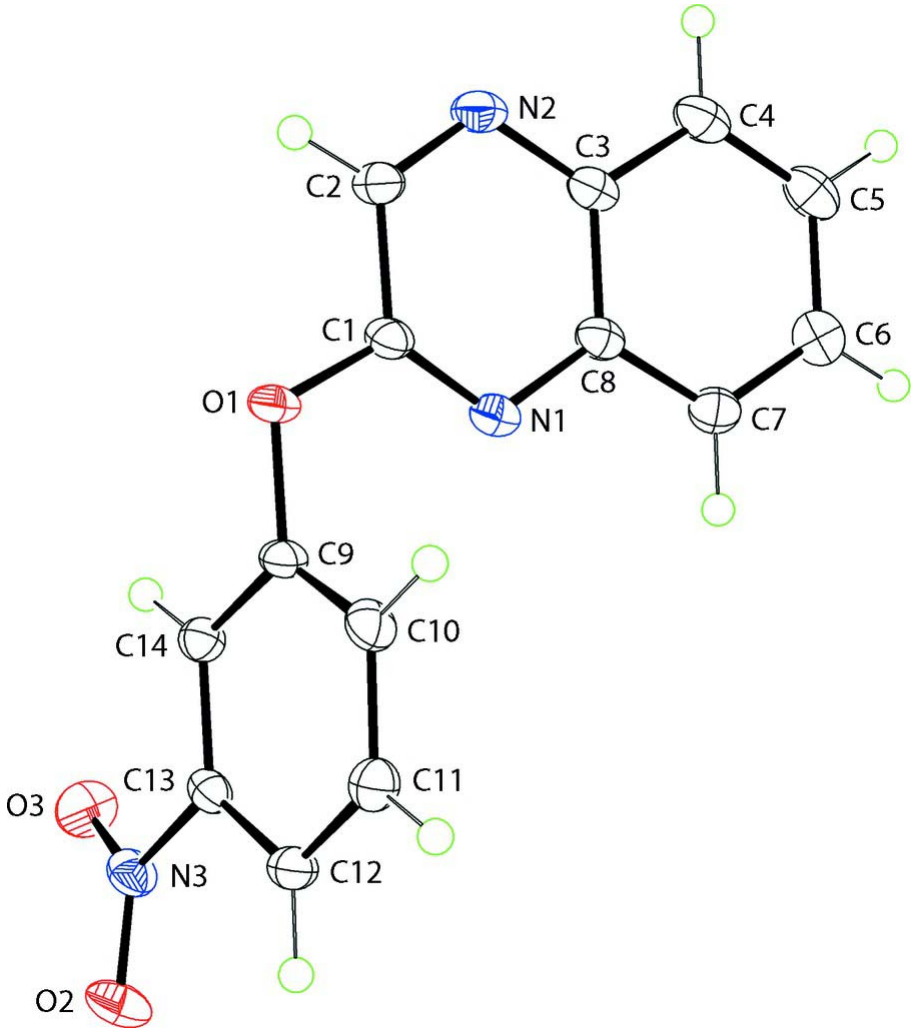

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

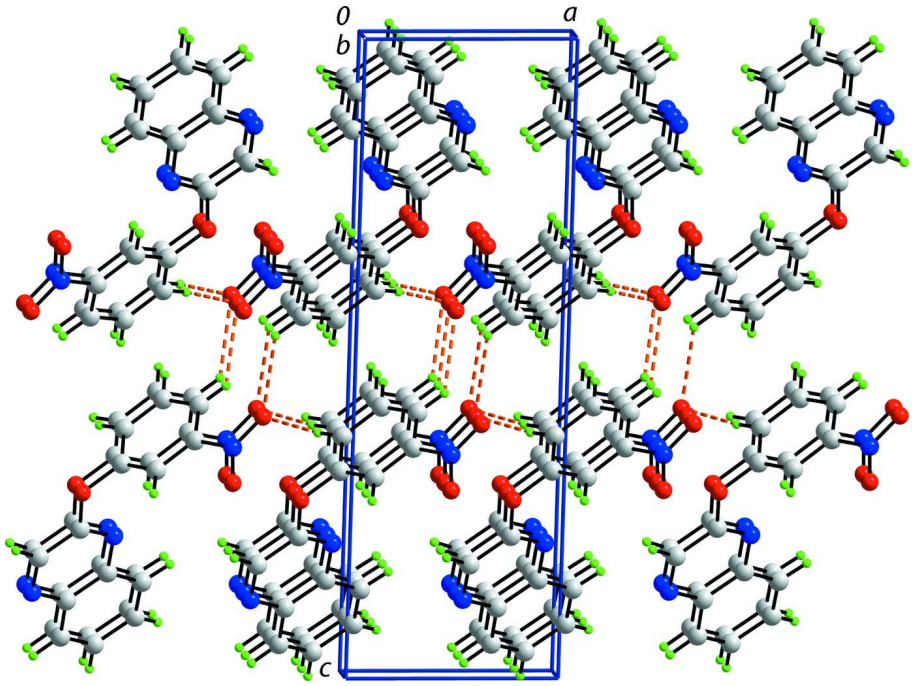

Figure 2

Supramolecular layer in the *ab* plane mediated by C–H···O interactions, shown as orange dashed lines, in (I).

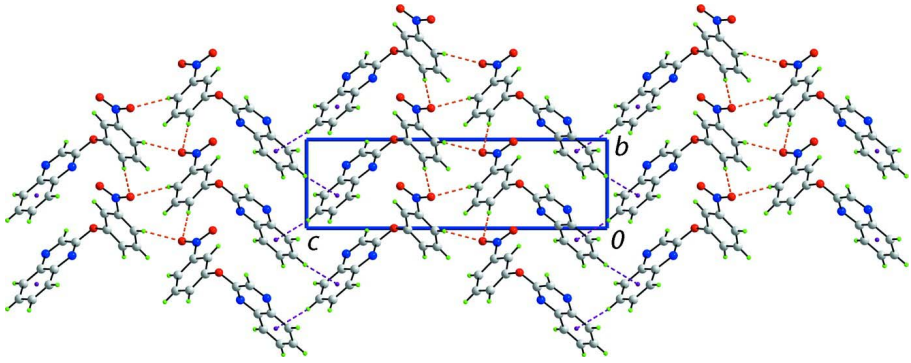

Figure 3

Unit-cell contents shown in projection down the *a* axis in (I), highlighting the stacking of layers along the *c* direction. The C–H···O and C–H···π interactions are shown as orange and purple dashed lines, respectively.

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Crystal data

$C_{14}H_9N_3O_3$
$M_r = 267.24$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.0643\ (6)$ Å
$b = 5.3676\ (5)$ Å
$c = 18.2443\ (17)$ Å
$\beta = 91.780\ (1)°$
$V = 593.58\ (10)$ Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2560 reflections
$\theta = 2.2–28.3°$
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Prism, red
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
5637 measured reflections
1502 independent reflections
1403 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.1°$
$h = -7 \rightarrow 7$
$k = -6 \rightarrow 6$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.093$
$S = 1.04$
1501 reflections
181 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
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.098P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Absolute structure: nd

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 $F^2 > 2\sigma(F^2)$ 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}
O1	1.2692 (2)	0.5001 (3)	0.29144 (7)	0.0241 (3)
O2	0.4391 (2)	0.8540 (4)	0.41409 (8)	0.0309 (4)
O3	0.5748 (2)	0.9758 (3)	0.31197 (9)	0.0309 (4)
N1	1.1038 (2)	0.1916 (4)	0.22012 (8)	0.0203 (4)
N2	1.4817 (3)	0.2107 (4)	0.13226 (9)	0.0242 (4)
N3	0.5762 (3)	0.8419 (4)	0.36598 (9)	0.0224 (4)
C1	1.2685 (3)	0.3411 (4)	0.23292 (10)	0.0201 (4)
C2	1.4591 (3)	0.3556 (4)	0.18864 (11)	0.0229 (4)
H2	1.5713	0.4735	0.2007	0.028*
C3	1.3147 (3)	0.0427 (4)	0.11749 (10)	0.0212 (4)
C4	1.3319 (3)	−0.1261 (5)	0.05877 (11)	0.0261 (5)
H4	1.4576	−0.1216	0.0290	0.031*
C5	1.1678 (3)	−0.2970 (5)	0.04435 (11)	0.0281 (5)
H5	1.1817	−0.4113	0.0050	0.034*
C6	0.9785 (3)	−0.3042 (5)	0.08741 (11)	0.0268 (5)
H6	0.8666	−0.4243	0.0774	0.032*
C7	0.9563 (3)	−0.1377 (5)	0.14384 (10)	0.0227 (4)

H7	0.8263	−0.1393	0.1716	0.027*
C8	1.1248 (3)	0.0355 (4)	0.16091 (10)	0.0194 (4)
C9	1.0867 (3)	0.4856 (4)	0.33663 (10)	0.0198 (4)
C10	1.0743 (3)	0.2998 (4)	0.38865 (11)	0.0228 (4)
H10	1.1866	0.1769	0.3930	0.027*
C11	0.8952 (3)	0.2945 (4)	0.43471 (11)	0.0229 (4)
H11	0.8867	0.1690	0.4712	0.027*
C12	0.7295 (3)	0.4715 (4)	0.42751 (10)	0.0200 (4)
H12	0.6051	0.4671	0.4579	0.024*
C13	0.7504 (3)	0.6547 (4)	0.37477 (10)	0.0181 (4)
C14	0.9282 (3)	0.6693 (4)	0.32869 (10)	0.0193 (4)
H14	0.9402	0.7989	0.2936	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0166 (6)	0.0277 (8)	0.0286 (7)	−0.0011 (6)	0.0085 (5)	−0.0071 (7)
O2	0.0254 (7)	0.0371 (9)	0.0307 (8)	0.0124 (7)	0.0081 (6)	−0.0029 (7)
O3	0.0265 (7)	0.0290 (9)	0.0372 (8)	0.0069 (7)	0.0003 (6)	0.0113 (7)
N1	0.0184 (7)	0.0230 (9)	0.0198 (7)	0.0034 (7)	0.0028 (6)	0.0005 (7)
N2	0.0211 (8)	0.0274 (10)	0.0244 (8)	0.0027 (7)	0.0074 (6)	0.0035 (8)
N3	0.0192 (7)	0.0220 (9)	0.0260 (8)	0.0050 (7)	0.0011 (6)	−0.0017 (8)
C1	0.0166 (8)	0.0204 (10)	0.0233 (9)	0.0038 (8)	0.0033 (7)	0.0004 (9)
C2	0.0188 (9)	0.0241 (11)	0.0262 (10)	0.0003 (9)	0.0056 (7)	0.0011 (9)
C3	0.0222 (9)	0.0231 (11)	0.0183 (9)	0.0061 (8)	0.0035 (7)	0.0025 (8)
C4	0.0285 (10)	0.0294 (12)	0.0208 (9)	0.0081 (10)	0.0062 (7)	0.0013 (9)
C5	0.0327 (11)	0.0308 (12)	0.0210 (9)	0.0080 (10)	0.0006 (8)	−0.0042 (9)
C6	0.0286 (10)	0.0272 (12)	0.0245 (10)	0.0019 (9)	−0.0024 (8)	−0.0005 (10)
C7	0.0227 (9)	0.0262 (11)	0.0193 (9)	0.0010 (9)	0.0017 (7)	0.0013 (9)
C8	0.0196 (8)	0.0211 (11)	0.0177 (8)	0.0045 (8)	0.0018 (6)	0.0026 (8)
C9	0.0134 (8)	0.0234 (10)	0.0228 (9)	−0.0010 (8)	0.0041 (6)	−0.0058 (9)
C10	0.0182 (9)	0.0205 (11)	0.0296 (10)	0.0048 (8)	−0.0001 (7)	−0.0018 (8)
C11	0.0244 (9)	0.0202 (11)	0.0240 (10)	0.0004 (8)	0.0016 (7)	0.0023 (8)
C12	0.0176 (8)	0.0228 (11)	0.0198 (9)	0.0003 (8)	0.0032 (6)	−0.0015 (8)
C13	0.0167 (8)	0.0181 (10)	0.0196 (8)	0.0025 (7)	0.0003 (6)	−0.0032 (8)
C14	0.0193 (9)	0.0202 (10)	0.0186 (8)	−0.0001 (8)	0.0014 (6)	−0.0007 (8)

Geometric parameters (Å, º)

O1—C1	1.367 (2)	C5—H5	0.9500
O1—C9	1.402 (2)	C6—C7	1.373 (3)
O2—N3	1.229 (2)	C6—H6	0.9500
O3—N3	1.219 (2)	C7—C8	1.410 (3)
N1—C1	1.297 (3)	C7—H7	0.9500
N1—C8	1.376 (3)	C9—C10	1.380 (3)
N2—C2	1.300 (3)	C9—C14	1.382 (3)
N2—C3	1.376 (3)	C10—C11	1.394 (3)
N3—C13	1.463 (2)	C10—H10	0.9500

C1—C2	1.433 (2)	C11—C12	1.386 (3)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.409 (3)	C12—C13	1.384 (3)
C3—C8	1.419 (2)	C12—H12	0.9500
C4—C5	1.373 (3)	C13—C14	1.390 (3)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.411 (3)		

C1—O1—C9	116.22 (15)	C6—C7—C8	120.51 (18)
C1—N1—C8	115.37 (16)	C6—C7—H7	119.7
C2—N2—C3	116.86 (16)	C8—C7—H7	119.7
O3—N3—O2	123.90 (18)	N1—C8—C7	119.41 (16)
O3—N3—C13	118.70 (16)	N1—C8—C3	121.23 (17)
O2—N3—C13	117.40 (17)	C7—C8—C3	119.35 (18)
N1—C1—O1	120.68 (16)	C10—C9—C14	122.35 (16)
N1—C1—C2	124.22 (18)	C10—C9—O1	120.34 (17)
O1—C1—C2	115.10 (17)	C14—C9—O1	117.25 (18)
N2—C2—C1	121.36 (19)	C9—C10—C11	119.30 (18)
N2—C2—H2	119.3	C9—C10—H10	120.3
C1—C2—H2	119.3	C11—C10—H10	120.3
N2—C3—C4	119.93 (17)	C12—C11—C10	120.34 (19)
N2—C3—C8	120.91 (17)	C12—C11—H11	119.8
C4—C3—C8	119.17 (18)	C10—C11—H11	119.8
C5—C4—C3	120.34 (18)	C13—C12—C11	118.07 (17)
C5—C4—H4	119.8	C13—C12—H12	121.0
C3—C4—H4	119.8	C11—C12—H12	121.0
C4—C5—C6	120.6 (2)	C12—C13—C14	123.39 (18)
C4—C5—H5	119.7	C12—C13—N3	118.84 (16)
C6—C5—H5	119.7	C14—C13—N3	117.76 (18)
C7—C6—C5	120.0 (2)	C9—C14—C13	116.51 (18)
C7—C6—H6	120.0	C9—C14—H14	121.7
C5—C6—H6	120.0	C13—C14—H14	121.7

C8—N1—C1—O1	−178.20 (17)	C4—C3—C8—N1	178.05 (18)
C8—N1—C1—C2	2.1 (3)	N2—C3—C8—C7	179.52 (18)
C9—O1—C1—N1	1.7 (3)	C4—C3—C8—C7	−0.8 (3)
C9—O1—C1—C2	−178.60 (17)	C1—O1—C9—C10	79.9 (2)
C3—N2—C2—C1	−0.3 (3)	C1—O1—C9—C14	−102.8 (2)
N1—C1—C2—N2	−1.8 (3)	C14—C9—C10—C11	0.5 (3)
O1—C1—C2—N2	178.42 (18)	O1—C9—C10—C11	177.72 (17)
C2—N2—C3—C4	−177.78 (19)	C9—C10—C11—C12	1.1 (3)
C2—N2—C3—C8	1.9 (3)	C10—C11—C12—C13	−1.5 (3)
N2—C3—C4—C5	178.96 (19)	C11—C12—C13—C14	0.3 (3)
C8—C3—C4—C5	−0.7 (3)	C11—C12—C13—N3	179.51 (17)
C3—C4—C5—C6	0.8 (3)	O3—N3—C13—C12	−167.50 (18)
C4—C5—C6—C7	0.7 (3)	O2—N3—C13—C12	12.5 (3)
C5—C6—C7—C8	−2.2 (3)	O3—N3—C13—C14	11.8 (3)
C1—N1—C8—C7	178.47 (18)	O2—N3—C13—C14	−168.20 (18)

C1—N1—C8—C3	−0.4 (3)	C10—C9—C14—C13	−1.7 (3)
C6—C7—C8—N1	−176.59 (19)	O1—C9—C14—C13	−178.92 (16)
C6—C7—C8—C3	2.3 (3)	C12—C13—C14—C9	1.3 (3)
N2—C3—C8—N1	−1.6 (3)	N3—C13—C14—C9	−177.99 (16)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C3–C8 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O2^{i}	0.95	2.34	3.282 (3)	173
C12—H12···O2^{ii}	0.95	2.44	3.159 (2)	133
C5—H5···*Cg*1^{iii}	0.95	2.99	3.696 (2)	133

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