Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Allyl-5-nitro-1*H*-benzimidazol-2(3*H*)-one

Younès Ouzidan,[a]* Youssef Kandri Rodi,[a] Adiba Kandri Rodi,[a] El Mokhtar Essassi,[b,c] Mohamed Saadi[d] and Lahcen El Ammari[d]

[a]Laboratoire de Chimie Organique Appliquée, Université Sidi Mohamed Ben Abdallah, Faculté des Sciences et Techniques, Route d'immouzzer, BP 2202 Fès, Morocco, [b]Laboratoire de Chimie Organique Hétérocyclique URAC21, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco, [c]INANOTECH (Institute of Nanomaterials and Nanotechnology), MAScIR, Av. de l'Armée Royale, Rabat, Morocco, and [d]Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco
Correspondence e-mail: ouzidan@yahoo.fr

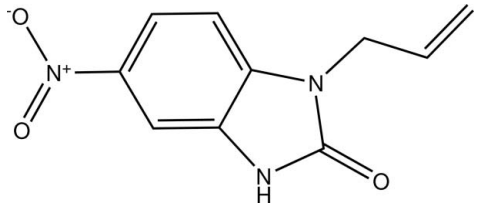
Received 18 February 2013; accepted 19 February 2013

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.168; data-to-parameter ratio = 13.4.

The benzimidazolone residue in the title molecule, $C_{10}H_9N_3O_3$, is almost planar, with the largest deviation from the mean plane being 0.016 (2) Å for the C atom linked to the nitro group. This plane is nearly perpendicular to the 1-allyl chain as indicated by the C—N—C—C torsion angle of 90.9 (3)°. The fused-ring system makes a dihedral angle of 5.6 (3)° with the nitro group, leading to a synperiplanar conformation. In the crystal, zigzag supramolecular chains are formed along the *a* axis by N—H···O hydrogen bonds.

Related literature

For pharmacological and biochemical properties of benzimidazoles and derivatives, see: Al Muhaimeed (1997); Scott *et al.* (2002); Nakano *et al.* (2000); Zhu *et al.* (2000); Zarrinmayeh *et al.* (1998). For related structures, see: Ouzidan *et al.* (2011*a*,*b*).



Experimental

Crystal data

$C_{10}H_9N_3O_3$ M_r = 219.20
Orthorhombic, *Pbca*
a = 8.3246 (3) Å
b = 14.9567 (6) Å
c = 16.4461 (7) Å
V = 2047.68 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.46 × 0.31 × 0.18 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
12145 measured reflections
1940 independent reflections
1483 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.168
S = 1.05
1940 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O3^{i}	0.86	1.96	2.801 (3)	164

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

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 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray data measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5200).

References

Al Muhaimeed, H. (1997). *J. Int. Med. Res.* **25**, 175–181.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Nakano, H., Inoue, T., Kawasaki, N., Miyataka, H., Matsumoto, H., Taguchi, T., Inagaki, N., Nagai, H. & Satoh, T. (2000). *Bioorg. Med. Chem.* **8**, 373–380.
Ouzidan, Y., Kandri Rodi, Y., Butcher, R. J., Essassi, E. M. & El Ammari, L. (2011*a*). *Acta Cryst.* E**67**, o283.
Ouzidan, Y., Kandri Rodi, Y., Saffon, N., Essassi, E. M. & Ng, S. W. (2011*b*). *Acta Cryst.* E**67**, o520.
Scott, L. J., Dunn, C. J., Mallarkey, G. & Sharpe, M. (2002). *Drugs*, **62**, 1503–1538.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zarrinmayeh, H., Nunes, A. M., Ornstein, P. L., Zimmerman, D. M., Arnold, M. B., Schober, D. A., Gackenheimer, S. L., Bruns, R. F., Hipskind, P. A., Britton, T. C., Cantrell, B. E. & Gehlert, D. R. (1998). *J. Med. Chem.* **41**, 2709–2719.
Zhu, Z., Lippa, B., Drach, J. C. & Townsend, L. B. (2000). *J. Med. Chem.* **43**, 2430–2437.

supplementary materials

Acta Cryst. (2013). E**69**, o431 [doi:10.1107/S1600536813004790]

1-Allyl-5-nitro-1*H*-benzimidazol-2(3*H*)-one

Younès Ouzidan, Youssef Kandri Rodi, Adiba Kandri Rodi, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

Comment

Benzimidazoles and their derivatives exhibit a number of important pharmacological properties, such as anti-histaminic (Al Muhaimeed, 1997) anti-ulcerative (Scott *et al.*, 2002) and anti-allergic (Nakano *et al.*, 2000). In addition, benzimidazole derivatives are effective against the human cytomegalovirus (HCMV) (Zhu *et al.*, 2000) and are also efficient selective neuropeptide Y Y1 receptor antagonists (Zarrinmayeh *et al.*, 1998).

As a continuation of our research work devoted to the development of substituted benzimidazol-2-one derivatives (Ouzidan *et al.*, 2011*a*, 2011*b*), we report in this paper the synthesis of a new benzimidazol-2-one derivative by action of allyl-bromide with 5-nitro-1*H*-benzo[*d*]imidazol-2(3*H*)one using similar conditions as employed in earlier studies.

The two fused five- and six-membered rings in the molecule of the title compound, $C_{10}H_9N_3O_3$, are approximately planar, the largest deviation from the mean plane being -0.016 (2) A° at C1 (Fig. 1). The dihedral angle between the benzimidazolone mean plane and the 1-allyl chain (C4—N3—C8—C9) is 90.9 (3)°. The fused-ring system makes a dihedral angle of 5.6 (3)° with the nitro group, leading to a syn-periplanar conformation. In the crystal, each molecule is linked to symmetry equivalents by N2—H2n···O3 hydrogen bonds, Table 2, forming a supramolecular zigzag chain running along the *a* axis, as shown in Fig. 2.

Experimental

To 5-Nitro-1*H*-benzo[*d*]imidazol-2(3*H*)-one (0.2 g, 1.11 mmol), potassium carbonate (0.3 g, 2.23 mmol), and tetra-*n*-butylammonium bromide (0.04 g, 0.11 mmol) in DMF (15 ml) was added allyl bromide (0.11 ml, 1.35 mmol). Stirring was continued at room temperature for 6 h. The salts were removed by filtration and the filtrate concentrated under reduced pressure. The residue was separated by chromatography on a column of silica gel with ethyl acetate/hexane (1/2) as eluent. Crystals were isolated when the solvent was allowed to evaporate.

Refinement

H atoms were located in a difference map and treated as riding with N—H = 0.86 Å, C—H = 0.93 Å (aromatic) and C—H = 0.97 Å (methylene), and with U_{iso}(H) = 1.2 U_{eq}(parent atom). Disorder is noted in the allyl group as evidence by the shorter than normal C9═C10 bond length of 1.177 (5) Å. Attempts to resolve this disorder for this room temperature data set were not successful.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

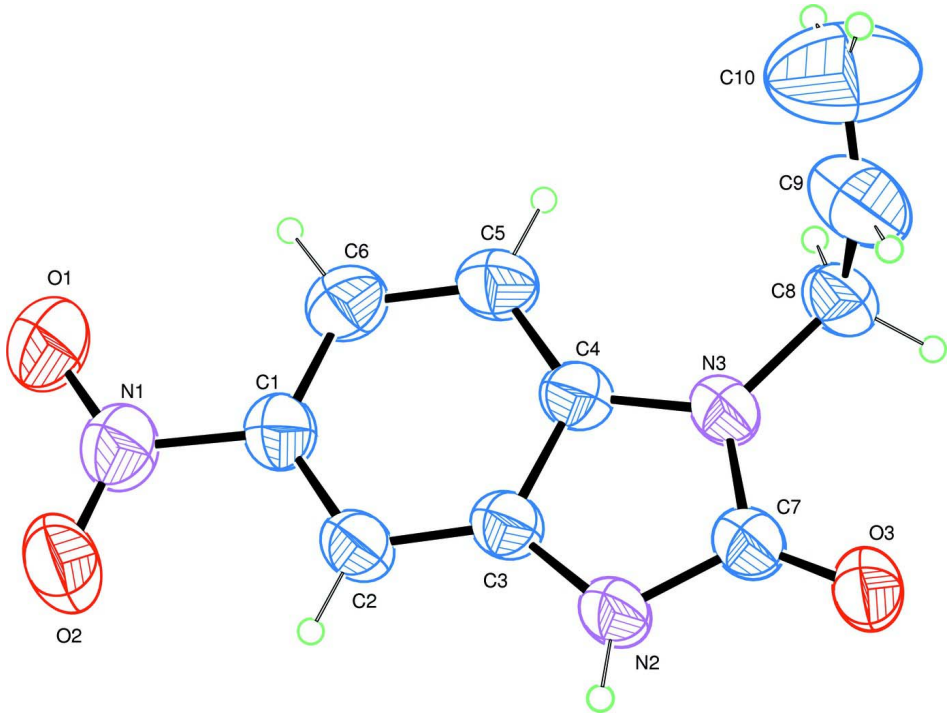


Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

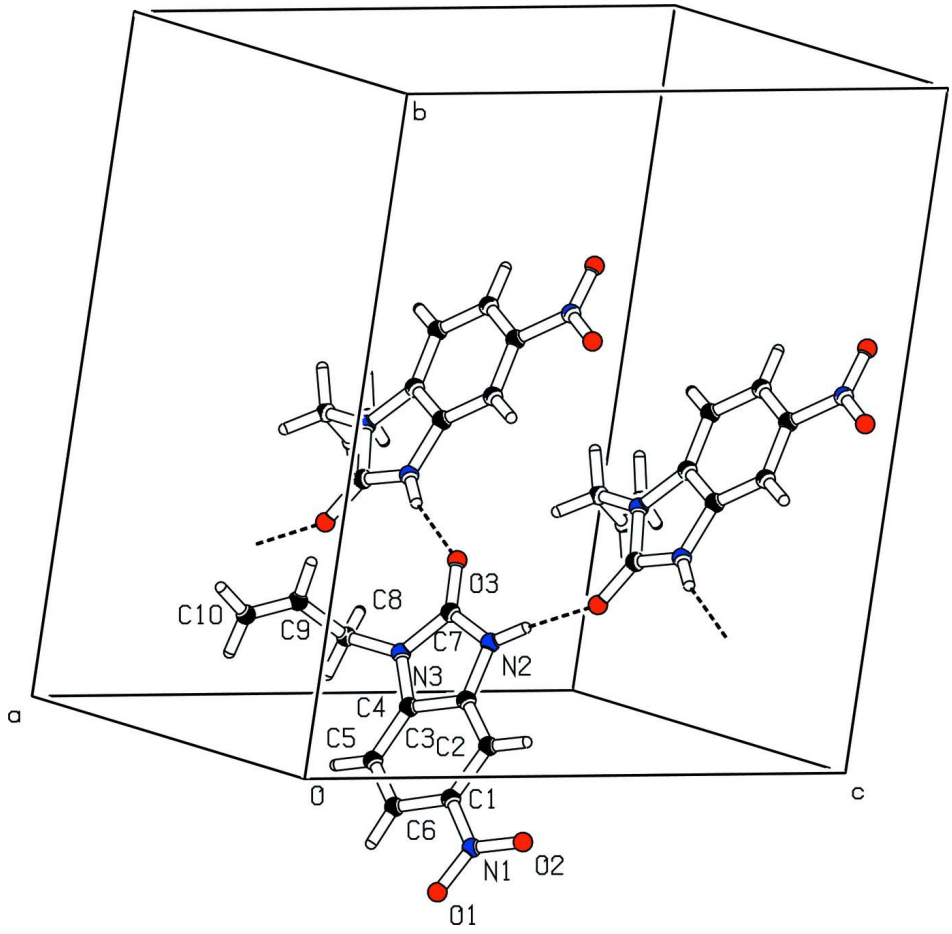


Figure 2

Molecule and its symmetry partner linked by N2—H2n···O3 hydrogen bonds. Symmetry codes: (i) 1/2 - *x*, 1 - *y*, 1/2 + *z*.

1-Allyl-5-nitro-1*H*-benzimidazol-2(3*H*)-one

Crystal data

$C_{10}H_9N_3O_3$
$M_r = 219.20$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 8.3246\ (3)$ Å
$b = 14.9567\ (6)$ Å
$c = 16.4461\ (7)$ Å
$V = 2047.68\ (14)$ Å^3
$Z = 8$
$F(000) = 912$
$D_x = 1.422$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1940 reflections
$\theta = 3.0$–$25.7°$
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Block, colourless
0.46 × 0.31 × 0.18 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
12145 measured reflections
1940 independent reflections
1483 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.7°$, $\theta_{min} = 3.0°$
$h = -10 \rightarrow 5$
$k = -17 \rightarrow 18$
$l = -20 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.168$
$S = 1.05$
1940 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 1.3415P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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	0.1801 (3)	−0.05403 (17)	0.37164 (15)	0.0479 (6)
C2	0.1529 (3)	0.02563 (16)	0.41211 (15)	0.0469 (6)
H2	0.0502	0.0439	0.4269	0.056*
C3	0.2866 (3)	0.07620 (16)	0.42913 (14)	0.0433 (6)
C4	0.4416 (3)	0.04786 (16)	0.40696 (14)	0.0432 (6)
C5	0.4658 (3)	−0.03268 (18)	0.36771 (16)	0.0517 (6)
H5	0.5685	−0.0516	0.3538	0.062*
C6	0.3328 (3)	−0.08415 (18)	0.34984 (16)	0.0532 (6)
H6	0.3449	−0.1387	0.3234	0.064*
C7	0.4612 (3)	0.18307 (17)	0.46777 (15)	0.0470 (6)
C8	0.7198 (3)	0.11726 (18)	0.41762 (15)	0.0505 (6)
H8A	0.7613	0.0567	0.4152	0.061*
H8B	0.7714	0.1474	0.4628	0.061*
C9	0.7608 (4)	0.1651 (3)	0.3401 (2)	0.0817 (10)
H9	0.7305	0.2249	0.3380	0.098*
C10	0.8257 (7)	0.1378 (4)	0.2815 (3)	0.1325 (19)
H10A	0.8593	0.0786	0.2794	0.159*
H10B	0.8434	0.1755	0.2375	0.159*
N1	0.0402 (3)	−0.10842 (16)	0.34996 (15)	0.0589 (6)
N2	0.3035 (2)	0.15796 (14)	0.46649 (13)	0.0506 (6)
H2N	0.2257	0.1889	0.4863	0.061*
N3	0.5459 (2)	0.11428 (13)	0.43179 (12)	0.0455 (5)
O1	0.0603 (3)	−0.17533 (15)	0.30908 (16)	0.0871 (8)
O2	−0.0927 (2)	−0.08426 (14)	0.37286 (15)	0.0746 (7)
O3	0.5177 (2)	0.25247 (13)	0.49549 (13)	0.0607 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0473 (13)	0.0443 (14)	0.0521 (13)	−0.0050 (11)	0.0021 (10)	−0.0018 (11)
C2	0.0375 (12)	0.0459 (14)	0.0573 (14)	0.0000 (10)	0.0064 (10)	−0.0019 (11)
C3	0.0391 (12)	0.0427 (13)	0.0479 (13)	0.0012 (10)	0.0048 (9)	−0.0019 (10)
C4	0.0395 (12)	0.0459 (13)	0.0441 (12)	0.0020 (10)	0.0034 (9)	−0.0012 (10)
C5	0.0453 (13)	0.0507 (15)	0.0592 (15)	0.0083 (11)	0.0064 (11)	−0.0064 (12)
C6	0.0573 (15)	0.0438 (14)	0.0584 (15)	0.0040 (11)	0.0039 (12)	−0.0081 (11)
C7	0.0381 (12)	0.0489 (14)	0.0541 (14)	−0.0002 (10)	0.0035 (10)	−0.0042 (11)
C8	0.0331 (12)	0.0633 (16)	0.0551 (14)	0.0048 (11)	0.0010 (10)	−0.0028 (12)
C9	0.0555 (17)	0.110 (3)	0.080 (2)	0.0014 (18)	0.0127 (16)	0.012 (2)
C10	0.181 (5)	0.122 (4)	0.094 (3)	0.021 (4)	0.050 (3)	0.018 (3)
N1	0.0592 (14)	0.0470 (13)	0.0704 (15)	−0.0068 (10)	0.0009 (11)	−0.0055 (11)
N2	0.0353 (10)	0.0469 (12)	0.0695 (13)	−0.0001 (8)	0.0088 (9)	−0.0147 (10)
N3	0.0332 (10)	0.0492 (12)	0.0542 (12)	−0.0002 (8)	0.0036 (8)	−0.0051 (9)
O1	0.0777 (15)	0.0703 (15)	0.1133 (18)	−0.0149 (11)	0.0075 (13)	−0.0401 (13)
O2	0.0483 (11)	0.0586 (13)	0.1169 (18)	−0.0083 (9)	0.0026 (11)	−0.0109 (11)
O3	0.0435 (9)	0.0561 (11)	0.0825 (13)	−0.0072 (8)	0.0022 (9)	−0.0194 (9)

Geometric parameters (Å, º)

C1—C2	1.383 (3)	C7—N2	1.365 (3)
C1—C6	1.396 (4)	C7—N3	1.381 (3)
C1—N1	1.465 (3)	C8—N3	1.467 (3)
C2—C3	1.374 (3)	C8—C9	1.501 (4)
C2—H2	0.9300	C8—H8A	0.9700
C3—N2	1.376 (3)	C8—H8B	0.9700
C3—C4	1.407 (3)	C9—C10	1.177 (5)
C4—N3	1.381 (3)	C9—H9	0.9300
C4—C5	1.381 (3)	C10—H10A	0.9300
C5—C6	1.380 (4)	C10—H10B	0.9300
C5—H5	0.9300	N1—O1	1.217 (3)
C6—H6	0.9300	N1—O2	1.223 (3)
C7—O3	1.228 (3)	N2—H2N	0.8600
C2—C1—C6	123.4 (2)	N3—C8—C9	111.9 (2)
C2—C1—N1	117.7 (2)	N3—C8—H8A	109.2
C6—C1—N1	118.8 (2)	C9—C8—H8A	109.2
C3—C2—C1	116.1 (2)	N3—C8—H8B	109.2
C3—C2—H2	122.0	C9—C8—H8B	109.2
C1—C2—H2	122.0	H8A—C8—H8B	107.9
C2—C3—N2	131.5 (2)	C10—C9—C8	129.3 (4)
C2—C3—C4	121.6 (2)	C10—C9—H9	115.3
N2—C3—C4	106.84 (19)	C8—C9—H9	115.3
N3—C4—C5	132.4 (2)	C9—C10—H10A	120.0
N3—C4—C3	106.5 (2)	C9—C10—H10B	120.0
C5—C4—C3	121.2 (2)	H10A—C10—H10B	120.0
C6—C5—C4	118.0 (2)	O1—N1—O2	122.5 (2)
C6—C5—H5	121.0	O1—N1—C1	118.8 (2)

C4—C5—H5	121.0	O2—N1—C1	118.7 (2)
C5—C6—C1	119.7 (2)	C7—N2—C3	110.48 (19)
C5—C6—H6	120.1	C7—N2—H2N	124.8
C1—C6—H6	120.1	C3—N2—H2N	124.8
O3—C7—N2	127.4 (2)	C7—N3—C4	109.99 (19)
O3—C7—N3	126.4 (2)	C7—N3—C8	123.3 (2)
N2—C7—N3	106.2 (2)	C4—N3—C8	126.5 (2)
C6—C1—C2—C3	1.2 (4)	C2—C1—N1—O2	−4.6 (4)
N1—C1—C2—C3	−178.0 (2)	C6—C1—N1—O2	176.2 (3)
C1—C2—C3—N2	178.7 (2)	O3—C7—N2—C3	179.0 (2)
C1—C2—C3—C4	−0.4 (4)	N3—C7—N2—C3	−1.3 (3)
C2—C3—C4—N3	179.1 (2)	C2—C3—N2—C7	−178.3 (3)
N2—C3—C4—N3	−0.2 (3)	C4—C3—N2—C7	0.9 (3)
C2—C3—C4—C5	−0.6 (4)	O3—C7—N3—C4	−179.1 (2)
N2—C3—C4—C5	−179.9 (2)	N2—C7—N3—C4	1.2 (3)
N3—C4—C5—C6	−178.8 (2)	O3—C7—N3—C8	−3.4 (4)
C3—C4—C5—C6	0.9 (4)	N2—C7—N3—C8	176.9 (2)
C4—C5—C6—C1	−0.1 (4)	C5—C4—N3—C7	179.1 (3)
C2—C1—C6—C5	−1.0 (4)	C3—C4—N3—C7	−0.6 (3)
N1—C1—C6—C5	178.2 (2)	C5—C4—N3—C8	3.5 (4)
N3—C8—C9—C10	−117.9 (5)	C3—C4—N3—C8	−176.1 (2)
C2—C1—N1—O1	174.6 (3)	C9—C8—N3—C7	−84.0 (3)
C6—C1—N1—O1	−4.6 (4)	C9—C8—N3—C4	90.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O3^{i}	0.86	1.96	2.801 (3)	164

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