Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[2-(1*H*-Benzimidazol-2-yl)ethyl]-1*H*-1,2,3-benzotriazole

Zhong Zhang,* Wei Lu and Difeng Wu

College of Chemistry and Chemical Engineering, Guangxi Normal University, Yucai Road 15, Guilin 541004, People's Republic of China
Correspondence e-mail: zhangzhong@mailbox.gxnu.edu.cn

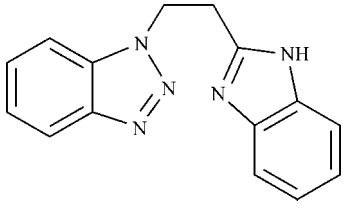
Received 4 October 2011; accepted 29 October 2011

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.125; data-to-parameter ratio = 12.7.

In the title compound, $C_{15}H_{13}N_5$, the N-containing heterocycles are linked by an ethylene spacer in a *gauche* conformation, the N—C—C—C torsion angle along the linker being 60.1 (3)°. The dihedral angle between the terminal benzotriazole and benzimidazole rings is 39.02 (6)°. In the crystal, adjacent molecules are connected by N—H···N hydrogen bonds, forming an infinite chain along the *c* axis. π–π stacking interactions [centroid–centroid distance = 3.8772 (7) Å] between the benzotriazole rings of neighbouring chains extend these chains into a supramolecular sheet in the *bc* plane. Weak intermolecular C—H···N interactions further stabilize the crystal structure.

Related literature

For the synthesis and antiviral activity of bis-heterocyclcic compounds containing both benzotriazole and benzimidazole, see: Pagani & Sparatore (1965); Paglietti *et al.* (1975); Katritzky *et al.* (1996); Yu *et al.* (2003); Tonelli *et al.* (2008). For the crystal structure of 1-(benzimidazol-2-ylmethyl)-1*H*-benzotriazole, see: Liu *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{13}N_5$
M_r = 263.30
Monoclinic, $P2_1/c$
a = 6.3510 (13) Å
b = 20.830 (4) Å
c = 9.901 (2) Å
β = 96.78 (3)°
V = 1300.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 K
0.37 × 0.32 × 0.26 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.969, T_{max} = 0.978
10985 measured reflections
2290 independent reflections
1608 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.125
S = 1.01
2290 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H14···N5^{i}	0.84	2.04	2.855 (3)	162
C1—H1···N3^{ii}	0.93	2.54	3.456 (3)	167

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

The authors are grateful for financial support from the Guangxi Science Foundation (grant No. 0832023) and the Scientific Research Foundation of Guangxi Normal University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2029).

References

Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2001). *SADABS*. Bruker AXS inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART*, *SAINT*. Bruker AXS inc., Madison, Wisconsin, USA.
Katritzky, A. R., Irina, D. A., Shcherbakova, I. A., Chem, J. & Belyakov, S. A. (1996). *J. Heterocycl. Chem.* **33**, 1107–1114.
Liu, S.-G., Ni, C.-L. & Li, Y.-Z. (2007). *Acta Cryst.* E**63**, o1827–o1828.
Pagani, F. & Sparatore, F. (1965). *Boll. Chim. Farm.* **104**, 427–431.
Paglietti, G., Boido, V. & Sparatore, F. (1975). *Farm. Ed. Sci.* **30**, 505–511.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tonelli, M., Paglietti, G., Boido, V., Sparatore, F., Marongiu, F., Marongiu, E., La Colla, P. & Loddo, R. (2008). *Chem. Biodivers.* **5**, 2386–2401.
Yu, K. L., Zhang, Y., Civiello, R. L., Kadow, K. F., Cianci, C., Krystal, M. & Meanwell, N. A. (2003). *Bioorg. Med. Chem. Lett.* **13**, 2141–2144.

supporting information

Acta Cryst. (2011). E**67**, o3167 [https://doi.org/10.1107/S1600536811045442]

1-[2-(1*H*-Benzimidazol-2-yl)ethyl]-1*H*-1,2,3-benzotriazole

Zhong Zhang, Wei Lu and Difeng Wu

S1. Comment

A family of asymmetric bis-heterocycle compounds comprising both benzotriazole and benzimidazole entities have been prepared (Pagani *et al.* 1965; Paglietti *et al.* 1975; Katritzky *et al.* 1996) and some of them exhibit potent antiviral activity (Yu *et al.* 2003; Tonelli *et al.* 2008), but the structure-function relationship of these novel potential drugs is still a research focus. The structural determination of 1-(Benzimidazol-2-ylmethyl)-1*H*-benzotriazole has been fulfilled (Liu *et al.* 2007). In this paper, the crystal structure of its analogue 1-(2-(1*H*-Benzimidazol-2-yl)ethyl)-1*H*-benzotriazole was reported. The molecular structure of the title compound, (I), is depicted in Fig. 1. In the molecule, benzotriazole and benzimidazole rings are arranged in a *gauche* conformation about the ethylidene linkage, as described by the N1—C7—C8—C9 torsion angle of 60.1 (3) °. Both heterocyclic rings are planar and the dihedral angle between them is 39.02 (6) °. In the crystal packing, adjacent molecules are self-assembled through intermolecular N4—H14···N5^{i} hydrogen bonds (Table 1) between benzimidazole groups into an infinite one-dimensional non-linear chain along the *c* axis (Fig. 2), which is further held together *via* face-to-face *π*—*π* stacking [centriod-centriod distance = 3.8772 (7) Å, symmetry code: -*x*, 1 - *y*,2 - *z*] between the benzotriazole groups coming from neighbouring non-linear chains resulting in a two-dimensional supramolecular layer parallel to the *bc* plane (Fig. 3). Weak C1—H1···N3^{ii} hydrogen bonds (Table 1) exist between the layers contributing to the construction of the three-dimensional network.

S2. Experimental

The mixture of 3-(1*H*-benzotriazole-1-yl)-propionic acid (3.8 g, 0.02 mol) and *o*-phenylenediamine (3.2 g, 0.03 mol) were dissolved in 2 mol/*L* HCl (10 ml) and refluxed for 10 h to yield a brown solution. After cooling, the solution was filtered to remove the unreacted *o*-phenylenediamine. Concentrated $NH_3.H_2O$ was added to the resulting filtrate continuously until the gray solid was precipitated completely. The gray solid was redissolved in a small amount of CH_3OH at room temperature. Two weeks ago, colorless block-like crystals of (I) suitable for X-ray diffraction analysis were obtained. (yield 47%) Elemental analysis found: C 68.69; H 5.32; N 26.64%; calculated for $C_{15}H_{13}N_5$: C 68.42; H 4.98; N 26.60%.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å and N—H = 0.84 Å) and refined as riding atoms, with U_{iso}(H) = 1.2 times U_{eq}(C) or 1.5 times U_{eq}(N).

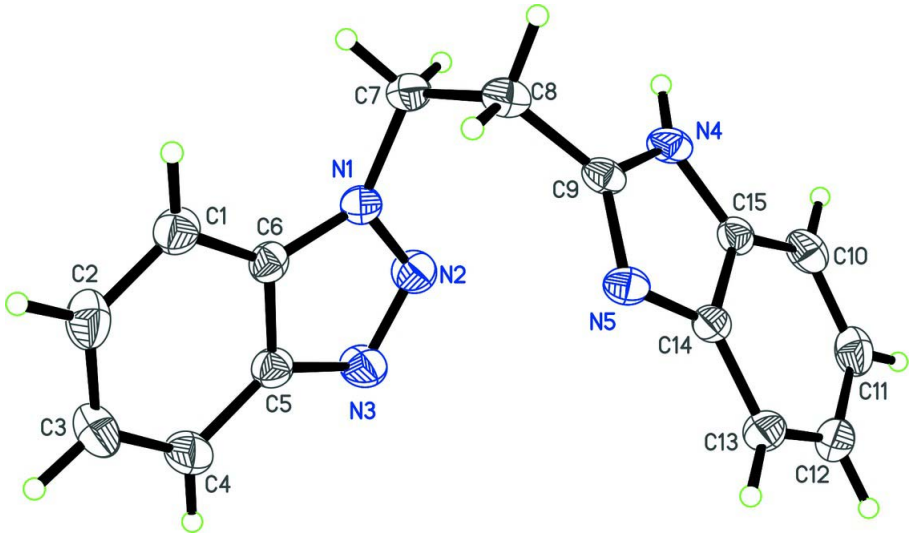


Figure 1

The molecular structure of the title compound (I), showing the atom labeling scheme and 30% displacement ellipsoids.

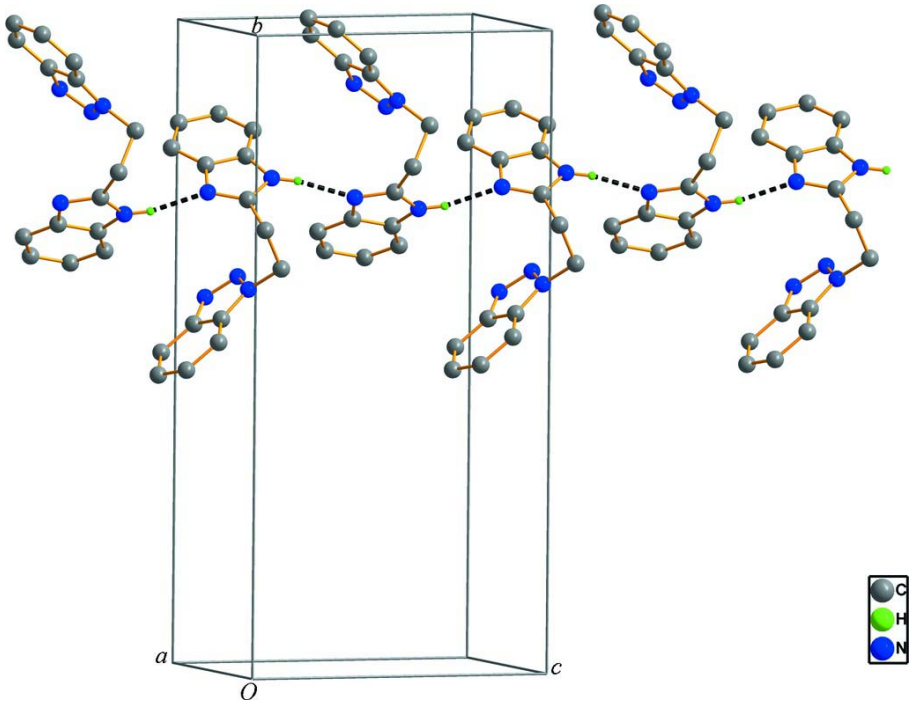


Figure 2

The crystal packing of the title compound (I), showing the one-dimensional hydrogen-bonding chain. Hydrogen bonds are shown as black dashed lines.

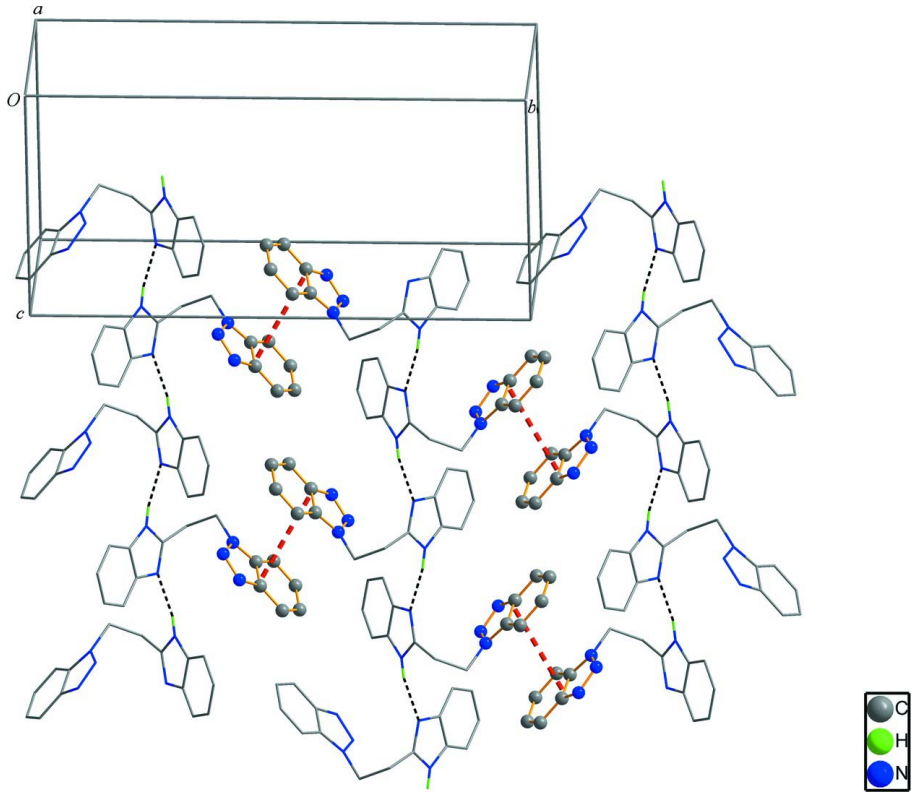


Figure 3

A view of the two-dimensional supramolecular sheet constructed from one-dimensional chains *via* π—π stacking of heterocyclic rings. π—π stacking interactions are represented by red dashed lines.

1-[2-(1*H*-Benzimidazol-2-yl)ethyl]-1*H*-1,2,3-benzotriazole

Crystal data

$C_{15}H_{13}N_5$
$M_r = 263.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.3510\ (13)$ Å
$b = 20.830\ (4)$ Å
$c = 9.901\ (2)$ Å
$\beta = 96.78\ (3)°$
$V = 1300.7\ (5)$ Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10162 reflections
$\theta = 3.2–27.8°$
$\mu = 0.09$ mm^{-1}
$T = 294$ K
Block, colourless
0.37 × 0.32 × 0.26 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.969$, $T_{max} = 0.978$
10985 measured reflections
2290 independent reflections
1608 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.2°$
$h = -7\rightarrow7$
$k = -24\rightarrow24$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.125$
$S = 1.01$
2290 reflections
181 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
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 0.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

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	−0.2105 (4)	0.96993 (13)	0.3185 (3)	0.0476 (7)
H1	−0.3426	0.9620	0.3468	0.057*
C2	−0.1838 (5)	1.01290 (14)	0.2171 (3)	0.0573 (8)
H2	−0.3016	1.0346	0.1748	0.069*
C3	0.0144 (5)	1.02507 (15)	0.1755 (3)	0.0567 (8)
H3	0.0252	1.0549	0.1068	0.068*
C4	0.1916 (5)	0.99482 (14)	0.2323 (3)	0.0539 (8)
H4	0.3230	1.0028	0.2029	0.065*
C5	0.1695 (4)	0.95122 (13)	0.3364 (3)	0.0414 (7)
C6	−0.0285 (4)	0.93888 (12)	0.3764 (3)	0.0367 (6)
C7	−0.1427 (4)	0.85935 (13)	0.5476 (3)	0.0448 (7)
H7A	−0.2662	0.8860	0.5547	0.054*
H7B	−0.0790	0.8493	0.6391	0.054*
C8	−0.2120 (4)	0.79717 (13)	0.4733 (3)	0.0435 (7)
H8A	−0.3149	0.7754	0.5220	0.052*
H8B	−0.2801	0.8074	0.3830	0.052*
C9	−0.0301 (4)	0.75382 (12)	0.4618 (2)	0.0344 (6)
C10	0.4117 (5)	0.65844 (13)	0.5972 (3)	0.0482 (7)
H10	0.4180	0.6501	0.6899	0.058*
C11	0.5649 (5)	0.63636 (13)	0.5222 (3)	0.0526 (8)
H11	0.6778	0.6127	0.5648	0.063*
C12	0.5546 (5)	0.64870 (13)	0.3839 (3)	0.0498 (8)
H12	0.6615	0.6333	0.3361	0.060*
C13	0.3915 (4)	0.68287 (13)	0.3161 (3)	0.0438 (7)
H13	0.3856	0.6904	0.2232	0.053*
C14	0.2348 (4)	0.70614 (12)	0.3900 (2)	0.0344 (6)
C15	0.2474 (4)	0.69362 (12)	0.5293 (2)	0.0363 (6)
N1	0.0085 (3)	0.89470 (10)	0.4772 (2)	0.0388 (6)

N2	0.2169 (4)	0.87999 (11)	0.4968 (2)	0.0491 (6)
N3	0.3165 (3)	0.91386 (12)	0.4137 (3)	0.0530 (7)
N4	0.0740 (3)	0.72406 (10)	0.5709 (2)	0.0398 (6)
H14	0.0439	0.7286	0.6512	0.060*
N5	0.0578 (3)	0.74388 (10)	0.3498 (2)	0.0379 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0375 (16)	0.0444 (17)	0.0615 (19)	−0.0022 (14)	0.0082 (14)	−0.0019 (15)
C2	0.0480 (19)	0.0470 (19)	0.074 (2)	0.0052 (15)	−0.0048 (16)	0.0073 (18)
C3	0.060 (2)	0.0481 (18)	0.061 (2)	−0.0064 (16)	0.0035 (17)	0.0156 (16)
C4	0.0446 (18)	0.0541 (19)	0.065 (2)	−0.0064 (15)	0.0167 (16)	0.0090 (17)
C5	0.0365 (15)	0.0413 (16)	0.0473 (17)	−0.0015 (13)	0.0086 (13)	−0.0024 (14)
C6	0.0373 (16)	0.0330 (15)	0.0399 (16)	−0.0013 (12)	0.0045 (13)	−0.0062 (13)
C7	0.0474 (17)	0.0458 (17)	0.0440 (17)	−0.0007 (14)	0.0173 (14)	−0.0020 (14)
C8	0.0403 (16)	0.0504 (17)	0.0411 (16)	−0.0085 (14)	0.0103 (13)	−0.0003 (14)
C9	0.0388 (15)	0.0400 (15)	0.0247 (14)	−0.0109 (12)	0.0045 (12)	−0.0014 (12)
C10	0.0574 (19)	0.0471 (17)	0.0390 (16)	−0.0050 (15)	0.0007 (15)	0.0113 (14)
C11	0.0498 (19)	0.0397 (17)	0.067 (2)	0.0042 (14)	0.0008 (16)	0.0082 (16)
C12	0.0539 (19)	0.0389 (17)	0.059 (2)	0.0011 (15)	0.0152 (16)	−0.0045 (15)
C13	0.0539 (18)	0.0435 (17)	0.0349 (15)	−0.0027 (14)	0.0092 (14)	−0.0063 (13)
C14	0.0454 (16)	0.0332 (14)	0.0249 (13)	−0.0068 (13)	0.0057 (12)	−0.0048 (12)
C15	0.0455 (16)	0.0321 (15)	0.0317 (14)	−0.0060 (13)	0.0063 (13)	−0.0012 (12)
N1	0.0346 (13)	0.0403 (13)	0.0422 (13)	−0.0008 (10)	0.0072 (10)	0.0007 (11)
N2	0.0369 (14)	0.0555 (16)	0.0547 (15)	0.0026 (12)	0.0045 (12)	0.0074 (13)
N3	0.0338 (13)	0.0617 (16)	0.0645 (17)	−0.0004 (12)	0.0099 (12)	0.0116 (14)
N4	0.0486 (14)	0.0480 (14)	0.0240 (12)	−0.0074 (11)	0.0095 (10)	−0.0008 (10)
N5	0.0419 (13)	0.0465 (13)	0.0257 (11)	−0.0022 (11)	0.0057 (10)	0.0004 (10)

Geometric parameters (Å, º)

C1—C2	1.370 (4)	C8—H8B	0.9700
C1—C6	1.388 (4)	C9—N5	1.316 (3)
C1—H1	0.9300	C9—N4	1.348 (3)
C2—C3	1.393 (4)	C10—C11	1.372 (4)
C2—H2	0.9300	C10—C15	1.383 (4)
C3—C4	1.353 (4)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.387 (4)
C4—C5	1.394 (4)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.366 (4)
C5—N3	1.376 (3)	C12—H12	0.9300
C5—C6	1.386 (3)	C13—C14	1.391 (3)
C6—N1	1.357 (3)	C13—H13	0.9300
C7—N1	1.453 (3)	C14—N5	1.391 (3)
C7—C8	1.529 (4)	C14—C15	1.397 (3)
C7—H7A	0.9700	C15—N4	1.375 (3)
C7—H7B	0.9700	N1—N2	1.350 (3)

C8—C9	1.481 (4)	N2—N3	1.303 (3)
C8—H8A	0.9700	N4—H14	0.8453

C2—C1—C6	116.0 (3)	N5—C9—N4	112.7 (2)
C2—C1—H1	122.0	N5—C9—C8	125.0 (2)
C6—C1—H1	122.0	N4—C9—C8	122.1 (2)
C1—C2—C3	122.0 (3)	C11—C10—C15	117.2 (3)
C1—C2—H2	119.0	C11—C10—H10	121.4
C3—C2—H2	119.0	C15—C10—H10	121.4
C4—C3—C2	121.9 (3)	C10—C11—C12	121.3 (3)
C4—C3—H3	119.0	C10—C11—H11	119.4
C2—C3—H3	119.0	C12—C11—H11	119.4
C3—C4—C5	117.3 (3)	C13—C12—C11	121.8 (3)
C3—C4—H4	121.3	C13—C12—H12	119.1
C5—C4—H4	121.3	C11—C12—H12	119.1
N3—C5—C6	108.4 (2)	C12—C13—C14	118.1 (3)
N3—C5—C4	131.2 (3)	C12—C13—H13	120.9
C6—C5—C4	120.4 (3)	C14—C13—H13	120.9
N1—C6—C5	104.6 (2)	C13—C14—N5	130.6 (2)
N1—C6—C1	133.1 (2)	C13—C14—C15	119.6 (2)
C5—C6—C1	122.3 (3)	N5—C14—C15	109.8 (2)
N1—C7—C8	111.6 (2)	N4—C15—C10	133.1 (2)
N1—C7—H7A	109.3	N4—C15—C14	104.8 (2)
C8—C7—H7A	109.3	C10—C15—C14	122.1 (3)
N1—C7—H7B	109.3	N2—N1—C6	109.9 (2)
C8—C7—H7B	109.3	N2—N1—C7	120.6 (2)
H7A—C7—H7B	108.0	C6—N1—C7	129.0 (2)
C9—C8—C7	111.8 (2)	N3—N2—N1	109.1 (2)
C9—C8—H8A	109.3	N2—N3—C5	108.0 (2)
C7—C8—H8A	109.3	C9—N4—C15	107.9 (2)
C9—C8—H8B	109.3	C9—N4—H14	124.0
C7—C8—H8B	109.3	C15—N4—H14	127.7
H8A—C8—H8B	107.9	C9—N5—C14	104.9 (2)

C6—C1—C2—C3	−0.5 (4)	N5—C14—C15—N4	0.5 (3)
C1—C2—C3—C4	0.5 (5)	C13—C14—C15—C10	−0.1 (4)
C2—C3—C4—C5	−0.9 (5)	N5—C14—C15—C10	−178.3 (2)
C3—C4—C5—N3	−179.5 (3)	C5—C6—N1—N2	−0.8 (3)
C3—C4—C5—C6	1.5 (4)	C1—C6—N1—N2	−179.3 (3)
N3—C5—C6—N1	0.4 (3)	C5—C6—N1—C7	−173.1 (2)
C4—C5—C6—N1	179.6 (2)	C1—C6—N1—C7	8.4 (5)
N3—C5—C6—C1	179.1 (2)	C8—C7—N1—N2	−83.6 (3)
C4—C5—C6—C1	−1.7 (4)	C8—C7—N1—C6	88.0 (3)
C2—C1—C6—N1	179.4 (3)	C6—N1—N2—N3	1.0 (3)
C2—C1—C6—C5	1.2 (4)	C7—N1—N2—N3	174.1 (2)
N1—C7—C8—C9	60.1 (3)	N1—N2—N3—C5	−0.7 (3)
C7—C8—C9—N5	−105.2 (3)	C6—C5—N3—N2	0.2 (3)
C7—C8—C9—N4	69.2 (3)	C4—C5—N3—N2	−178.9 (3)

C15—C10—C11—C12	−0.2 (4)	N5—C9—N4—C15	1.9 (3)
C10—C11—C12—C13	−0.4 (4)	C8—C9—N4—C15	−173.2 (2)
C11—C12—C13—C14	0.8 (4)	C10—C15—N4—C9	177.2 (3)
C12—C13—C14—N5	177.2 (3)	C14—C15—N4—C9	−1.4 (3)
C12—C13—C14—C15	−0.5 (4)	N4—C9—N5—C14	−1.5 (3)
C11—C10—C15—N4	−177.9 (3)	C8—C9—N5—C14	173.4 (2)
C11—C10—C15—C14	0.5 (4)	C13—C14—N5—C9	−177.3 (3)
C13—C14—C15—N4	178.7 (2)	C15—C14—N5—C9	0.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H14···N5^{i}	0.84	2.04	2.855 (3)	162
C1—H1···N3^{ii}	0.93	2.54	3.456 (3)	167

Symmetry codes: (i) x, $-y+3/2$, $z+1/2$; (ii) $x-1$, y, z.