3-Ethyl-4-[3-(1H-imidazol-1-yl)propyl]-5-phenyl-4H-1,2,4-triazole dihydrate

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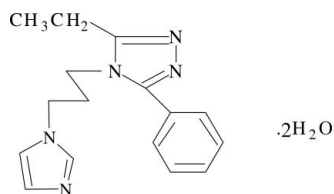Correspondence e-mail: vasuki.arasi@yahoo.com

In the title compound, $C_{16}H_{19}N_5\cdot 2H_2O$, the triazole ring makes dihedral angles of 70.61 (6) and 41.89 (8)°, respectively, with the imidazole and benzene rings. The water molecules are involved in intermolecular O—H⋯N and O—H⋯O hydrogen bonds, which stabilize the crystal packing.

Related literature

For a related structure, see: Rizzoli *et al.* (2009); Kalkan *et al.* (2007). For bond lengths and angles in triazole rings, see: Thenmozhi *et al.* (2010); Rizzoli *et al.* (2009); Dolzhenko *et al.* (2010); Ocak İskeleli *et al.* (2005); Ünver *et al.* (2010). For the biological activity of triazole Schiff bases, see: Thenmozhi *et al.* (2010) and of 1,2,4-triazole derivatives, see: Ünver *et al.* (2010). For the search for and synthesis of new antibiotics, see: Köysal *et al.* (2006). For the synthesis, see: Ünver *et al.* (2009).

Experimental

Crystal data

$C_{16}H_{19}N_5\cdot 2H_2O$	V = 1715.0 (3) Å³
M_r = 317.39	Z = 4
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
a = 11.0787 (16) Å	μ = 0.68 mm⁻¹
b = 9.8428 (8) Å	T = 293 K
c = 16.3289 (18) Å	0.30 × 0.20 × 0.20 mm
β = 105.602 (9)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	2868 independent reflections
Absorption correction: ψ scan North *et al.* (1968)	2166 reflections with $I > 2\sigma(I)$
T_{min} = 0.821, T_{max} = 0.876	R_{int} = 0.029
3030 measured reflections	2 standard reflections every 60 min intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.123	
S = 1.08	
2868 reflections	$\Delta\rho_{max}$ = 0.19 e Å⁻³
226 parameters	$\Delta\rho_{min}$ = −0.18 e Å⁻³
6 restraints	

Table 1
Selected geometric parameters (Å, °).

C7—N1	1.308 (2)	C8—N3	1.363 (2)
C7—N3	1.373 (2)	N1—N2	1.387 (2)
C8—N2	1.312 (2)		

C8—N3—C7	105.09 (14)	C7—N3—C11	128.05 (14)
C8—N3—C11	126.52 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1A⋯O2	0.91 (1)	1.98 (1)	2.882 (3)	172 (3)
O1—H1B⋯N2ⁱ	0.90 (1)	2.02 (1)	2.913 (2)	170 (3)
O2—H2A⋯N5ⁱⁱ	0.91 (1)	1.95 (1)	2.859 (3)	176 (2)
O2—H2B⋯O1ⁱⁱⁱ	0.91 (1)	2.08 (2)	2.949 (3)	160 (3)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2213).

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supporting information

3-Ethyl-4-[3-(1*H*-imidazol-1-yl)propyl]-5-phenyl-4*H*-1,2,4-triazole dihydrate

Anuradha Gurumoorthy, Vasuki Gopalsamy, Dilek Ünlüer, Esra Düğdü and Babu Varghesee

S1. Comment

The biological importance of imidazoles and triazoles has stimulated much work on these heterocycles. 1,2,4-Triazole is a basic aromatic ring and possesses good coordination ability due to the presence of nitrogen atoms (Thenmozhi *et al.*,2010). 1,2,4-Triazole compounds posses important pharmacology activities such as antifungal and antiviral activities. Examples of such compounds bearing the 1,2,4-Triazole residues are fluconazole, the powerful azole antifungal agent as well as the potent antiviral N-nucleoside ribavirin. Furthermore various 1,2,4-Triazole derivatives have been reported as fungicidal, insecticidal, antimicrobial as well as anticonvulsants, antidepressants and plant growth regulator anticoagulants (Ünver *et al.*, 2010). 1,2,4-Triazole derivatives are also used to build polymetallic complexes. Compounds derived from triazole possess antimicrobial, analgesic, anti-inflammatory, local anesthetic, antineoplastic and antimalarial properties. Some triazole Schiff bases also exhibit antiproliferative and anticancer activities (Thenmozhi *et al.*, 2010). 1,2,4-Triazole moieties interact strongly with heme iron and aromatic substituents on the triazoles are very effective for interacting with the active site of aromatase. Furthermore, it was reported that compounds having triazole moieties such as Vorozole, Anastrozole and Letrozole appear to be very effective aromatese inhibitors very useful for preventing breast cancer (Ünver *et al.*, 2010). Some of the azole derivatives used as common antibiotics, such as amphotericin B, exhibit toxic effects on humans along with antimicrobial effects. Although different antimicrobial agents are used in the treatment of microbial infections, an increasing resistance to these drugs is observed. Therefore, the search for and synthesis of new antibiotics different from commonly used ones is of current importance (Köysal *et al.*, 2006). In a search for new triazole compounds with better biological activity, the title compound (I), was synthesized. We report here the crystal structure of the title compound, (I) (Fig.1), a new 1,2,4-triazole derivative. The compound (I) crystallizes as a dihydrate, the bond lengths and angles (Table 1) are generally normal in the triazole ring (Thenmozhi *et al.*, 2010; Rizzoli *et al.*, 2009; Dolzhenko *et al.*, 2010; Ocak Ískeleli *et al.*, 2005; Ünver *et al.*, 2010). Atom N3 has a trigonal configuration, the sum of the three bond angles around them being 360° (Kalkan *et al.*, 2007). The dihedral angles between the planes A(N1/N2/C7/N3/C8), B(N4/C14/N5/C15/C16) and C(C1/C2/C3/C4/C5/C6) are A/B = 70.61 (6)°, A/C = 41.89 (8)° and B/C = 68.16 (7)°. The triazole ring is essentially planar with r.m.s deviation of 0.0046Å (Dolzhenko *et al.*, 2010). The C—N bond lengths in the triazole ring of all molecules lie in the range of 1.260 (3)–1.349 (4) Å. These are longer than a typical double C=N bond [1.269 (2) Å], but shorter than a C—N single bond [1.443 (4) Å], (Thenmozhi *et al.*, 2010) indicating the possibility of electron delocalization. The N3–C11–C12–C13 torsion angle of 172.00 (15)° indicates that the triazole ring and the imidazole moiety has an E-configuration across the C11–C12 bond. This configuration is stabilized by an intramolecular C11–H11···N4 hydrogen bond with H11···N4 distance of 2.54 Å. The uncoordinated water molecules are involved in intermolecular O–H···O and O–H···N hydrogen bonds (Table 2), which stabilize the crystal packing.

S2. Experimental

The compound was synthesized by published method (Ünver *et al.*, 2009).

S3. Refinement

Water H atoms were located in a difference Fourier map and isotropically refined with O—H distance restraints of 0.90 (1) Å. All the other H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93Å (aromatic) and 0.97Å (methylene), N—H = 0.86 Å, and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}$ or $1.5U_{eq}$(parent atom). In the absence of significant anomalous scattering effects, Friedel pairs were merged.
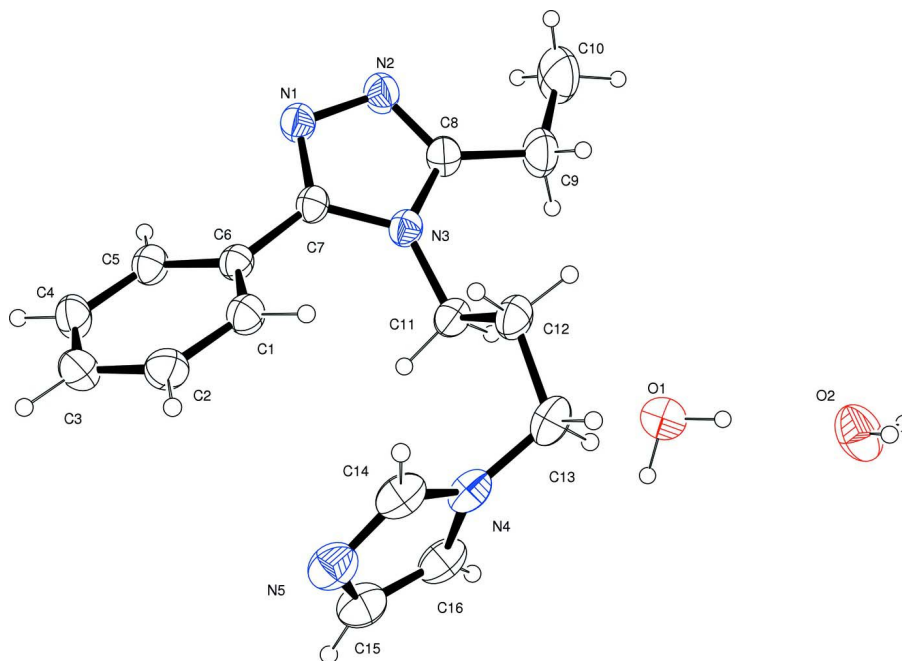
Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.
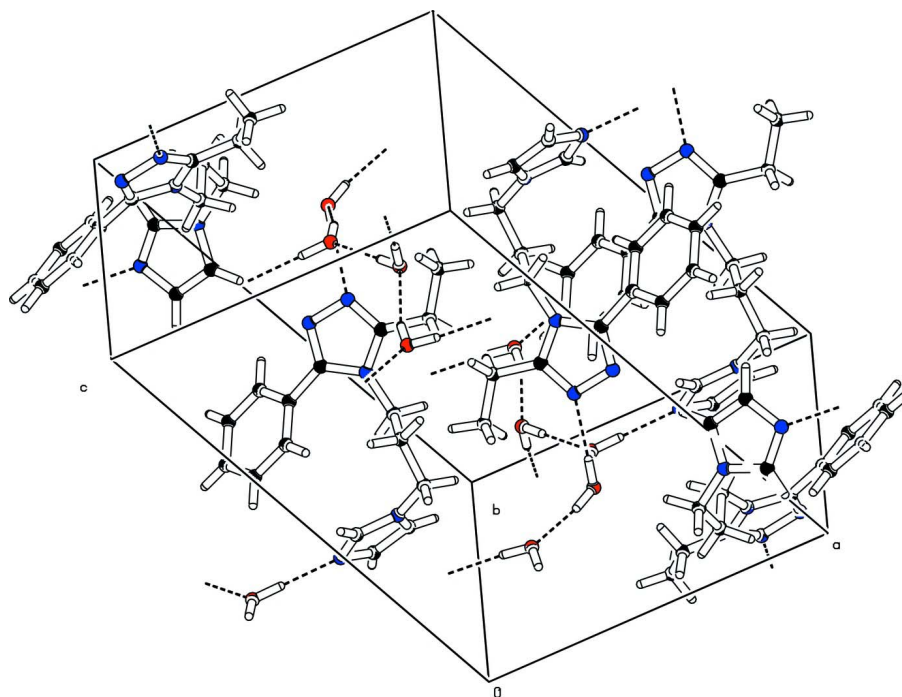
Figure 2

Crystal packing of the title compound viewed along the a axiS. Intermolecular O—H···O and O—H···N hydrogen bonds are shown as dashed lines.

Figure 3

Crystal Packing of the title compound with hydrogen bonds.

3-Ethyl-4-[3-(1H-imidazol-1-yl)propyl]-5-phenyl-4H-1,2,4-triazole dihydrate

Crystal data

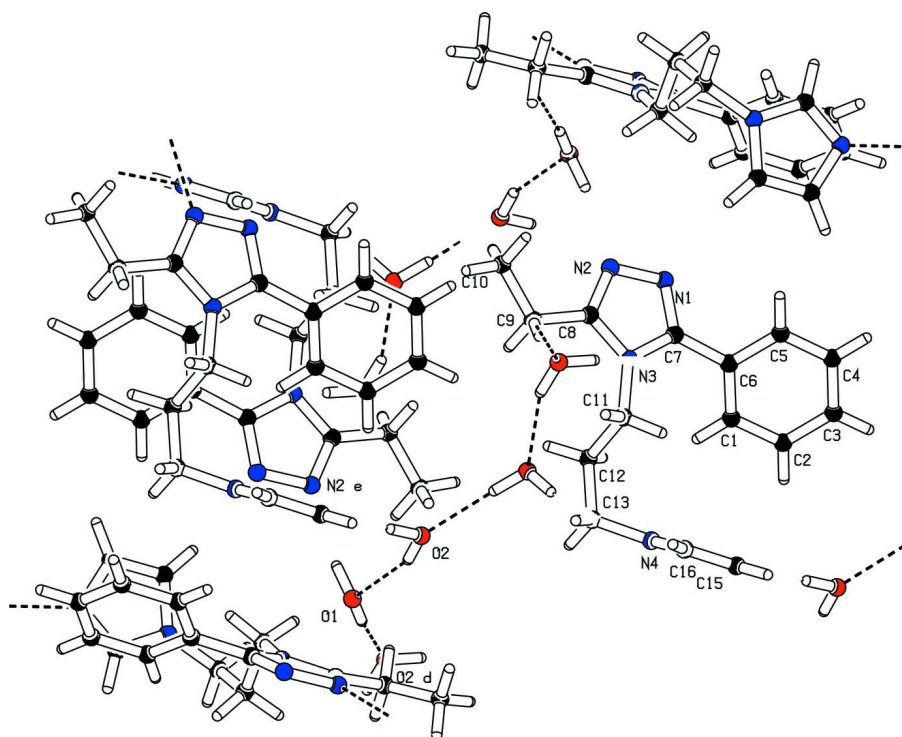|	
$C_{16}H_{19}N_5 \cdot 2H_2O$	$F(000) = 680$
$M_r = 317.39$	$D_x = 1.229$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
$a = 11.0787$ (16) Å	Cell parameters from 25 reflections
$b = 9.8428$ (8) Å	$\theta = 20–30°$
$c = 16.3289$ (18) Å	$\mu = 0.68$ mm^{-1}
$\beta = 105.602$ (9)°	$T = 293$ K
$V = 1715.0$ (3) Å^3	Block, colourless
$Z = 4$	$0.30 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 Diffractometer	2166 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.029$
Graphite monochromator	$\theta_{max} = 64.9°$, $\theta_{min} = 4.1°$
$\omega–2\tau$ scan	$h = 0 \rightarrow 13$
Absorption correction: ψ scan	$k = 0 \rightarrow 11$
North *et al.* (1968)	$l = -19 \rightarrow 18$
$T_{min} = 0.821$, $T_{max} = 0.876$	2 standard reflections every 60 min
3030 measured reflections	intensity decay: none
2868 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from
Least-squares matrix: full	neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent
$wR(F^2) = 0.123$	and constrained refinement
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.2107P]$
2868 reflections	where $P = (F_o^2 + 2F_c^2)/3$
226 parameters	$(\Delta/\sigma)_{max} < 0.001$
6 restraints	$\Delta\rho_{max} = 0.19$ e Å^{-3}
Primary atom site location: structure-invariant	$\Delta\rho_{min} = -0.18$ e Å^{-3}
direct methods	Extinction correction: *SHELXL*,
Secondary atom site location: difference Fourier	Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
map	Extinction coefficient: 0.0246 (12)

Special details

Experimental. Number of psi-scan sets used was 5 Theta correction was applied. Averaged transmission function was used. No Fourier smoothing was applied.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	1.10619 (16)	0.09615 (18)	0.08555 (11)	0.0519 (4)

H1	1.0554	0.0218	0.0878	0.062*
C2	1.23425 (18)	0.0845 (2)	0.11690 (12)	0.0609 (5)
H2	1.2692	0.0026	0.1400	0.073*
C3	1.31051 (18)	0.1935 (2)	0.11417 (12)	0.0655 (6)
H3	1.3970	0.1855	0.1354	0.079*
C4	1.25803 (18)	0.3152 (2)	0.07964 (12)	0.0623 (5)
H4	1.3094	0.3893	0.0779	0.075*
C5	1.13010 (17)	0.32727 (17)	0.04779 (11)	0.0509 (4)
H5	1.0956	0.4092	0.0242	0.061*
C6	1.05207 (15)	0.21742 (16)	0.05067 (10)	0.0429 (4)
C7	0.91650 (15)	0.23322 (16)	0.01343 (10)	0.0430 (4)
C8	0.71432 (16)	0.23090 (19)	−0.00821 (11)	0.0529 (5)
C9	0.58650 (18)	0.2091 (3)	0.00312 (15)	0.0772 (6)
H9A	0.5689	0.1124	0.0005	0.093*
H9B	0.5856	0.2406	0.0592	0.093*
C10	0.4853 (2)	0.2794 (3)	−0.06124 (19)	0.1100 (10)
H10A	0.5027	0.3750	−0.0600	0.165*
H10B	0.4066	0.2646	−0.0486	0.165*
H10C	0.4813	0.2441	−0.1167	0.165*
C11	0.83527 (17)	0.11825 (17)	0.12819 (10)	0.0496 (4)
H11A	0.7754	0.1576	0.1551	0.060*
H11B	0.9185	0.1359	0.1648	0.060*
C12	0.81484 (18)	−0.03413 (18)	0.12166 (11)	0.0571 (5)
H12A	0.8667	−0.0732	0.0884	0.069*
H12B	0.7279	−0.0529	0.0925	0.069*
C13	0.8473 (2)	−0.0997 (2)	0.20926 (13)	0.0658 (5)
H13A	0.7918	−0.0643	0.2411	0.079*
H13B	0.8335	−0.1969	0.2029	0.079*
C14	1.0814 (2)	−0.1225 (2)	0.23923 (13)	0.0618 (5)
H14	1.0804	−0.1814	0.1945	0.074*
C15	1.1436 (2)	0.0064 (2)	0.34639 (14)	0.0700 (6)
H15	1.1959	0.0543	0.3912	0.084*
C16	1.0180 (2)	0.0092 (2)	0.32597 (12)	0.0655 (5)
H16	0.9685	0.0584	0.3532	0.079*
N1	0.86819 (13)	0.29858 (15)	−0.05759 (9)	0.0524 (4)
N2	0.73910 (14)	0.29737 (16)	−0.07142 (10)	0.0572 (4)
N3	0.82282 (12)	0.18678 (13)	0.04670 (8)	0.0455 (4)
N4	0.97689 (15)	−0.07477 (15)	0.25699 (9)	0.0564 (4)
N5	1.18406 (17)	−0.07686 (18)	0.29187 (11)	0.0692 (5)
O1	0.60569 (16)	0.10016 (18)	0.26136 (10)	0.0807 (5)
O2	0.43707 (15)	−0.11055 (19)	0.28415 (13)	0.0920 (6)
H1A	0.554 (2)	0.030 (2)	0.2642 (16)	0.127 (11)*
H1B	0.650 (2)	0.121 (3)	0.3147 (9)	0.134 (11)*
H2A	0.3579 (13)	−0.096 (2)	0.2882 (17)	0.105 (9)*
H2B	0.444 (2)	−0.2016 (12)	0.277 (2)	0.154 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0520 (10)	0.0489 (10)	0.0551 (10)	−0.0035 (8)	0.0146 (8)	0.0050 (8)
C2	0.0562 (11)	0.0673 (12)	0.0575 (11)	0.0090 (9)	0.0124 (8)	0.0122 (9)
C3	0.0467 (11)	0.0881 (15)	0.0583 (11)	−0.0024 (10)	0.0086 (8)	−0.0020 (10)
C4	0.0537 (11)	0.0680 (12)	0.0668 (12)	−0.0186 (9)	0.0190 (9)	−0.0075 (10)
C5	0.0543 (11)	0.0451 (9)	0.0560 (10)	−0.0047 (8)	0.0193 (8)	−0.0015 (8)
C6	0.0464 (10)	0.0417 (8)	0.0423 (8)	−0.0029 (7)	0.0152 (7)	−0.0023 (7)
C7	0.0455 (9)	0.0376 (8)	0.0483 (9)	−0.0039 (7)	0.0169 (7)	−0.0005 (7)
C8	0.0472 (10)	0.0528 (10)	0.0598 (10)	−0.0034 (8)	0.0163 (8)	0.0020 (8)
C9	0.0505 (12)	0.0929 (16)	0.0929 (15)	−0.0052 (11)	0.0271 (11)	0.0124 (13)
C10	0.0514 (14)	0.151 (3)	0.131 (2)	0.0045 (15)	0.0297 (14)	0.031 (2)
C11	0.0578 (10)	0.0464 (9)	0.0489 (9)	−0.0042 (8)	0.0218 (8)	−0.0010 (7)
C12	0.0665 (12)	0.0479 (10)	0.0615 (11)	−0.0122 (8)	0.0252 (9)	−0.0007 (8)
C13	0.0780 (14)	0.0530 (11)	0.0757 (13)	−0.0041 (10)	0.0366 (11)	0.0124 (10)
C14	0.0839 (15)	0.0492 (10)	0.0594 (11)	0.0147 (10)	0.0314 (11)	0.0078 (9)
C15	0.0886 (16)	0.0605 (12)	0.0631 (12)	0.0040 (11)	0.0240 (11)	0.0049 (10)
C16	0.0951 (16)	0.0532 (11)	0.0590 (11)	0.0097 (10)	0.0394 (11)	0.0041 (9)
N1	0.0461 (8)	0.0548 (9)	0.0562 (8)	−0.0011 (7)	0.0137 (6)	0.0098 (7)
N2	0.0439 (9)	0.0624 (9)	0.0638 (9)	−0.0005 (7)	0.0121 (7)	0.0096 (8)
N3	0.0477 (8)	0.0424 (7)	0.0485 (7)	−0.0045 (6)	0.0163 (6)	0.0002 (6)
N4	0.0741 (11)	0.0474 (8)	0.0559 (8)	0.0066 (7)	0.0314 (8)	0.0092 (7)
N5	0.0786 (12)	0.0630 (11)	0.0704 (10)	0.0143 (9)	0.0278 (9)	0.0130 (9)
O1	0.0767 (11)	0.0899 (12)	0.0706 (10)	−0.0057 (9)	0.0114 (8)	0.0019 (8)
O2	0.0619 (10)	0.0805 (12)	0.1268 (15)	0.0036 (8)	0.0135 (9)	−0.0038 (10)

Geometric parameters (Å, º)

C1—C2	1.377 (2)	C11—N3	1.465 (2)
C1—C6	1.387 (2)	C11—C12	1.516 (2)
C1—H1	0.9300	C11—H11A	0.9700
C2—C3	1.373 (3)	C11—H11B	0.9700
C2—H2	0.9300	C12—C13	1.522 (3)
C3—C4	1.384 (3)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C4—C5	1.377 (3)	C13—N4	1.459 (2)
C4—H4	0.9300	C13—H13A	0.9700
C5—C6	1.393 (2)	C13—H13B	0.9700
C5—H5	0.9300	C14—N5	1.307 (3)
C6—C7	1.469 (2)	C14—N4	1.352 (2)
C7—N1	1.308 (2)	C14—H14	0.9300
C7—N3	1.373 (2)	C15—C16	1.341 (3)
C8—N2	1.312 (2)	C15—N5	1.371 (3)
C8—N3	1.363 (2)	C15—H15	0.9300
C8—C9	1.492 (3)	C16—N4	1.372 (2)
C9—C10	1.485 (3)	C16—H16	0.9300
C9—H9A	0.9700	N1—N2	1.387 (2)

C9—H9B	0.9700	O1—H1A	0.906 (10)
C10—H10A	0.9600	O1—H1B	0.902 (10)
C10—H10B	0.9600	O2—H2A	0.909 (9)
C10—H10C	0.9600	O2—H2B	0.910 (10)
C2—C1—C6	120.80 (16)	N3—C11—H11A	108.6
C2—C1—H1	119.6	C12—C11—H11A	108.6
C6—C1—H1	119.6	N3—C11—H11B	108.6
C3—C2—C1	120.29 (18)	C12—C11—H11B	108.6
C3—C2—H2	119.9	H11A—C11—H11B	107.6
C1—C2—H2	119.9	C11—C12—C13	111.13 (15)
C2—C3—C4	119.64 (18)	C11—C12—H12A	109.4
C2—C3—H3	120.2	C13—C12—H12A	109.4
C4—C3—H3	120.2	C11—C12—H12B	109.4
C5—C4—C3	120.35 (18)	C13—C12—H12B	109.4
C5—C4—H4	119.8	H12A—C12—H12B	108.0
C3—C4—H4	119.8	N4—C13—C12	112.37 (15)
C4—C5—C6	120.35 (17)	N4—C13—H13A	109.1
C4—C5—H5	119.8	C12—C13—H13A	109.1
C6—C5—H5	119.8	N4—C13—H13B	109.1
C1—C6—C5	118.56 (16)	C12—C13—H13B	109.1
C1—C6—C7	122.80 (14)	H13A—C13—H13B	107.9
C5—C6—C7	118.59 (15)	N5—C14—N4	112.54 (18)
N1—C7—N3	110.00 (14)	N5—C14—H14	123.7
N1—C7—C6	123.22 (14)	N4—C14—H14	123.7
N3—C7—C6	126.76 (14)	C16—C15—N5	110.43 (19)
N2—C8—N3	110.02 (15)	C16—C15—H15	124.8
N2—C8—C9	125.15 (17)	N5—C15—H15	124.8
N3—C8—C9	124.82 (17)	C15—C16—N4	106.56 (17)
C10—C9—C8	113.94 (19)	C15—C16—H16	126.7
C10—C9—H9A	108.8	N4—C16—H16	126.7
C8—C9—H9A	108.8	C7—N1—N2	107.35 (13)
C10—C9—H9B	108.8	C8—N2—N1	107.52 (13)
C8—C9—H9B	108.8	C8—N3—C7	105.09 (14)
H9A—C9—H9B	107.7	C8—N3—C11	126.52 (14)
C9—C10—H10A	109.5	C7—N3—C11	128.05 (14)
C9—C10—H10B	109.5	C14—N4—C16	105.73 (17)
H10A—C10—H10B	109.5	C14—N4—C13	127.14 (17)
C9—C10—H10C	109.5	C16—N4—C13	127.00 (17)
H10A—C10—H10C	109.5	C14—N5—C15	104.73 (18)
H10B—C10—H10C	109.5	H1A—O1—H1B	108.4 (14)
N3—C11—C12	114.56 (14)	H2A—O2—H2B	106.5 (14)
C6—C1—C2—C3	0.1 (3)	C6—C7—N3—C8	177.38 (15)
C1—C2—C3—C4	−0.1 (3)	N1—C7—N3—C11	−174.85 (15)
C2—C3—C4—C5	−0.3 (3)	C6—C7—N3—C11	3.7 (3)
C3—C4—C5—C6	0.6 (3)	C12—C11—N3—C8	84.7 (2)
C2—C1—C6—C5	0.2 (3)	C12—C11—N3—C7	−102.89 (19)

C2—C1—C6—C7	177.55 (16)	N5—C14—N4—C16	0.8 (2)
C4—C5—C6—C1	−0.5 (2)	N5—C14—N4—C13	176.98 (15)
C4—C5—C6—C7	−177.98 (15)	C15—C16—N4—C14	−0.6 (2)
C1—C6—C7—N1	−137.55 (18)	C15—C16—N4—C13	−176.81 (16)
C5—C6—C7—N1	39.8 (2)	C12—C13—N4—C14	−67.3 (2)
C1—C6—C7—N3	44.1 (2)	C12—C13—N4—C16	108.1 (2)
C5—C6—C7—N3	−138.58 (17)	N4—C14—N5—C15	−0.6 (2)
N2—C8—C9—C10	−4.5 (3)	C16—C15—N5—C14	0.2 (2)
N3—C8—C9—C10	174.1 (2)	C7—N3—C11—C12	−102.89 (19)
N3—C11—C12—C13	172.00 (15)	C6—C7—N3—C11	3.7 (3)
C11—C12—C13—N4	−58.7 (2)	C6—C7—N3—C8	177.38 (15)
N5—C15—C16—N4	0.3 (2)	N1—C7—N3—C11	−174.85 (15)
N3—C7—N1—N2	0.85 (18)	N2—C8—N3—C11	174.88 (15)
C6—C7—N1—N2	−177.79 (14)	C9—C8—N3—C11	−3.9 (3)
N3—C8—N2—N1	−0.6 (2)	C5—C6—C7—N1	39.8 (2)
C9—C8—N2—N1	178.21 (19)	N1—C7—C6—C1	−137.55 (18)
C7—N1—N2—C8	−0.15 (19)	N3—C7—C6—C1	44.1 (2)
N2—C8—N3—C7	1.09 (19)	N2—C8—C9—C10	−4.5 (3)
C9—C8—N3—C7	−177.73 (19)	C10—C9—C8—N3	174.1 (2)
N2—C8—N3—C11	174.88 (15)	N4—C13—C12—C11	−58.7 (2)
C9—C8—N3—C11	−3.9 (3)	N3—C11—C12—C13	172.00 (15)
N1—C7—N3—C8	−1.19 (18)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2	0.91 (1)	1.98 (1)	2.882 (3)	172 (3)
O1—H1B···N2ⁱ	0.90 (1)	2.02 (1)	2.913 (2)	170 (3)
O2—H2A···N5ⁱⁱ	0.91 (1)	1.95 (1)	2.859 (3)	176 (2)
O2—H2B···O1ⁱⁱⁱ	0.91 (1)	2.08 (2)	2.949 (3)	160 (3)

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