organic compounds

1,4-Bis[1-(pyridin-4-ylmethyl)-1H-benzimidazol-2-yl]butane dihydrate

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The title compound, $C_{30}H_{28}N_6 \cdot 2H_2O$, is an example of a new N-donor neutral organic ligand. The asymmetric unit consists of two half-molecules (organic) and two water molecules; each independent organic molecule is centrosymmetric. The water molecules are linked by O—H···O hydrogen bonds, forming infinite chains stretching parallel to the a axis. These water chains also link the molecules of the title compound via C—H···O and O—H···N hydrogen bonds, generating a two-dimensional supramolecular layer-like structure.

Related literature

For related literature about coordination polymers, see: Leininger *et al.* (2000). For related literature about flexible neutral N-donor ligands related to the title compound and their ability to form helical and interpenetrating structures, see: Chesnut *et al.* (1999); Chu *et al.* (2007); Liu & Tilley (1997); Lo *et al.* (2000); Tong *et al.* (1999). For the synthesis of 1,4-bis(benzimidazol-2-yl)butane, see: (Chen *et al.* (2002).
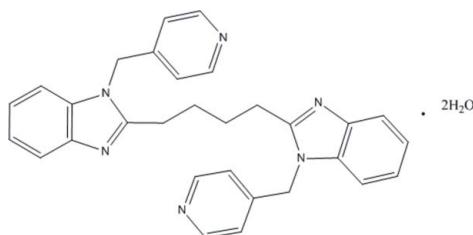
Experimental

Crystal data

$C_{30}H_{28}N_6 \cdot 2H_2O$	V = 2628.2 (8) Å^3
M_r = 508.62	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 4.9430 (2) Å	μ = 0.08 mm^{-1}
b = 27.969 (5) Å	T = 293 (2) K
c = 19.042 (5) Å	0.35 × 0.32 × 0.28 mm
β = 93.295 (5)°	

Data collection

Bruker APEX CCD area-detector diffractometer	16155 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	6112 independent reflections
T_{min} = 0.971, T_{max} = 0.977	2470 reflections with $I > 2\sigma(I)$
	R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.137	
S = 1.00	
6112 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
355 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
6 restraints	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1A···N5^i	0.86 (2)	2.01 (2)	2.854 (3)	171 (3)
O1W—H1B···O2W	0.83 (2)	1.97 (2)	2.800 (3)	177 (3)
O2W—H2B···N6^{ii}	0.88 (2)	2.04 (2)	2.858 (3)	154 (3)
O2W—H2A···O1W^{iii}	0.85 (2)	1.93 (2)	2.779 (3)	178 (3)
C13—H13···O2W	0.93	2.53	3.390 (4)	155

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2075).

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supplementary materials

1,4-Bis[1-(pyridin-4-ylmethyl)-1*H*-benzimidazol-2-yl]butane dihydrate

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Comment

The research on coordination polymers exhibiting novel structures and properties is an attractive field (Leininger *et al.*, 2000). The key factor for construction of coordination polymers is the selection of the organic ligand. Among various organic ligands, flexible N-donor ligands are beneficial when trying to form helical and interpenetrating structures (Chesnut *et al.*, 1999; Chu *et al.*, 2007; Liu & Tilley 1997; Lo *et al.*, 2000; Tong *et al.*, 1999). In this paper, we describe the isolation and structural characterization of a new N-donor neutral ligand, 1,4-bis(1-(pyridin-4-ylmethyl)-1*H*-benzo[*d*]imidazol-2-yl)butane (hereafter *L*).

The asymmetric part of the unit cell contains two crystallographically independent *L* molecules located each on an inversion center and two water molecules (Fig. 1). The butyl groups in different *L* molecules display TTT (T = *trans*) conformations. The dihedral angles between the phenyl and pyridine rings are 82.2 and 106.2°, respectively, in the two different *L* molceules. The water molecules are linked by O—H⋯O bonds to form an infinite water chain along the *a* axis. In addition, the water molecules in the water chains donate hydrogen bonds to N atoms of *L* molecules, and *L* molecules donate C—H⋯O bonds to water molecules. So the water chains link all *L* molecules along the *c* axis to generate a two dimensional supramolecular layerlike structure (Fig. 2). The sheet is waved and parallel to the *a,c* plane (Fig. 3).

Experimental

A mixture of 1,4-bis(benzimidazol-2-yl)butane (Chen *et al.*, 2002) (2.90 g, 10 mmol) and NaOH (0.80 g, 20 mmol) in DMSO (30 ml) was stirred at 60°C for 1 h, then 4-(chloromethyl)pyridine (5.10 g, 40 mmol) was added. The mixture was cooled to room temperature after stirring at 60°C for 24 h, and then poured into 200 ml of water. A colorless solid of *L* formed immediately, which was isolated by filtration in 65% yield after drying in air. Crystals suitable for X-ray diffraction were grown from 70% ethanol.

Refinement

All H atoms on C atoms were poisitioned geometrically and refined as riding atoms, with C—H = 0.93–0.97 Å, and U_{iso} = 1.2 or 1.5 U_{eq} (C). The H atoms of the water molecule were located in a difference Fourier map and then refined isotropically.

Figures
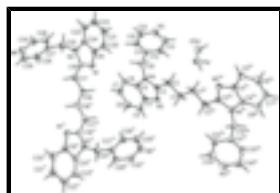
Fig. 1. A view of the title molecule. Displacement ellipsoids are drawn at the 30% probability level.
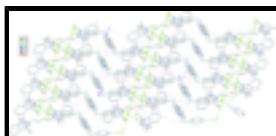
Fig. 2. Symmetry codes: (iv) $3 - x$, $1 - y$, $2 - z$; (v) $2 - x$, $1 - y$, $1 - z$.
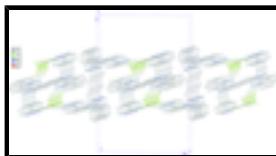
Fig. 3. and 3. View of two dimensional supramolecular layerlike strucutre of the title molecule along different directions. Green dashed lines represent O—H···O hydrogen bonds within the water chains, gray dashed lines represent of C—H···O bonds. All H atoms except H1A, H1B and H13 are omitted for clarity.

1,4-Bis[1-(pyridin-4-ylmethyl)-1H-benzimidazol-2-yl]butane dihydrate

Crystal data

$C_{30}H_{28}N_6 \cdot 2H_2O$	$Z = 4$
$M_r = 508.62$	$F_{000} = 1080$
Monoclinic, $P2_1/n$	$D_x = 1.285$ Mg m^{-3}
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 4.9430$ (2) Å	$\theta = 1.8–28.3°$
$b = 27.969$ (5) Å	$\mu = 0.08$ mm^{-1}
$c = 19.042$ (5) Å	$T = 293$ (2) K
$\beta = 93.295$ (5)°	Block, colorless
$V = 2628.2$ (8) Å^3	$0.35 \times 0.32 \times 0.28$ mm

Data collection

Bruker APEX CCD area-detector diffractometer	6112 independent reflections
Radiation source: fine-focus sealed tube	2470 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.065$
$T = 293(2)$ K	$\theta_{max} = 28.3°$
ω scans	$\theta_{min} = 1.8°$
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	$h = -6 \rightarrow 6$
$T_{min} = 0.971$, $T_{max} = 0.977$	$k = -34 \rightarrow 35$
16155 measured reflections	$l = -24 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.137$	$w = 1/[\sigma^2(F_o^2) + (0.040P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$

6112 reflections

$\Delta\rho_{max} = 0.16$ e Å^{-3}

355 parameters

$\Delta\rho_{min} = -0.24$ e Å^{-3}

6 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.8933 (5)	0.50658 (8)	0.47192 (11)	0.0472 (6)
H1	0.9561	0.5336	0.4453	0.057*
H2	0.7298	0.5163	0.4939	0.057*
C2	0.8277 (5)	0.46522 (8)	0.42202 (12)	0.0486 (7)
H3	0.9911	0.4562	0.3995	0.058*
H4	0.7709	0.4380	0.4491	0.058*
C3	0.6115 (5)	0.47634 (9)	0.36662 (12)	0.0444 (6)
C4	0.2902 (5)	0.51006 (9)	0.30361 (12)	0.0468 (6)
C5	0.0874 (6)	0.54023 (10)	0.27606 (13)	0.0584 (7)
H5	0.0467	0.5687	0.2983	0.070*
C6	−0.0516 (6)	0.52635 (11)	0.21446 (15)	0.0687 (8)
H6	−0.1882	0.5459	0.1948	0.082*
C7	0.0090 (6)	0.48374 (11)	0.18125 (14)	0.0662 (8)
H7	−0.0877	0.4757	0.1396	0.079*
C8	0.2062 (6)	0.45308 (10)	0.20766 (13)	0.0573 (7)
H8	0.2455	0.4246	0.1852	0.069*
C9	0.3437 (5)	0.46709 (9)	0.27001 (12)	0.0455 (6)
C10	0.6533 (5)	0.39772 (9)	0.30030 (13)	0.0566 (7)
H10A	0.8251	0.3940	0.3270	0.068*
H10B	0.6853	0.3935	0.2509	0.068*
C11	0.4576 (5)	0.35964 (9)	0.32270 (14)	0.0518 (7)
C12	0.3915 (6)	0.35553 (10)	0.39164 (16)	0.0730 (9)
H12	0.4690	0.3762	0.4253	0.088*
C13	0.2124 (7)	0.32119 (11)	0.41118 (16)	0.0839 (10)
H13	0.1733	0.3192	0.4583	0.101*
C14	0.1546 (7)	0.29493 (11)	0.29927 (18)	0.0778 (9)
H14	0.0726	0.2740	0.2665	0.093*

C15	0.3333 (6)	0.32844 (10)	0.27557 (15)	0.0662 (8)
H15	0.3689	0.3298	0.2282	0.079*
C16	0.4202 (5)	0.47921 (8)	0.01279 (13)	0.0568 (7)
H16A	0.2286	0.4860	0.0046	0.068*
H16B	0.4571	0.4759	0.0631	0.068*
C17	0.4827 (6)	0.43240 (9)	−0.02227 (13)	0.0630 (8)
H17A	0.4378	0.4353	−0.0723	0.076*
H17B	0.6759	0.4264	−0.0160	0.076*
C18	0.3344 (6)	0.39059 (9)	0.00522 (13)	0.0533 (7)
C19	0.0803 (6)	0.34537 (10)	0.06411 (13)	0.0563 (7)
C20	−0.1044 (6)	0.32642 (12)	0.10847 (15)	0.0758 (9)
H20	−0.2057	0.3462	0.1361	0.091*
C21	−0.1343 (7)	0.27759 (14)	0.11065 (16)	0.0844 (10)
H21	−0.2585	0.2643	0.1399	0.101*
C22	0.0181 (7)	0.24777 (12)	0.06990 (18)	0.0843 (10)
H22	−0.0031	0.2148	0.0734	0.101*
C23	0.1984 (6)	0.26558 (11)	0.02472 (16)	0.0736 (9)
H23	0.2973	0.2456	−0.0033	0.088*
C24	0.2264 (6)	0.31486 (10)	0.02284 (14)	0.0555 (7)
C25	0.5705 (6)	0.32965 (9)	−0.06941 (14)	0.0639 (8)
H25A	0.6288	0.2971	−0.0595	0.077*
H25B	0.7302	0.3499	−0.0666	0.077*
C26	0.2227 (6)	0.30358 (10)	−0.16366 (16)	0.0681 (8)
H26	0.1473	0.2829	−0.1319	0.082*
C27	0.4421 (6)	0.33192 (9)	−0.14281 (14)	0.0549 (7)
C28	0.5399 (6)	0.36186 (10)	−0.19213 (16)	0.0721 (9)
H28	0.6857	0.3819	−0.1804	0.087*
C29	0.4197 (8)	0.36214 (12)	−0.25995 (18)	0.0842 (10)
H29	0.4892	0.3828	−0.2927	0.101*
C30	0.1172 (7)	0.30636 (11)	−0.23204 (18)	0.0759 (9)
H30	−0.0305	0.2871	−0.2450	0.091*
N1	0.1506 (5)	0.39272 (8)	0.05213 (11)	0.0595 (6)
N2	0.3876 (4)	0.34486 (7)	−0.01566 (11)	0.0540 (6)
N3	0.0923 (5)	0.29067 (9)	0.36614 (16)	0.0804 (8)
N4	0.5492 (4)	0.44589 (7)	0.31133 (10)	0.0459 (5)
N5	0.4621 (4)	0.51524 (7)	0.36370 (10)	0.0469 (5)
N6	0.2121 (6)	0.33468 (10)	−0.28065 (13)	0.0762 (7)
O1W	0.5470 (4)	0.39740 (7)	0.55905 (10)	0.0684 (6)
O2W	0.0482 (4)	0.35252 (8)	0.57538 (11)	0.0784 (6)
H1A	0.550 (6)	0.4250 (7)	0.5783 (16)	0.118*
H1B	0.401 (5)	0.3840 (10)	0.5656 (17)	0.118*
H2A	−0.106 (5)	0.3658 (11)	0.5693 (15)	0.118*
H2B	0.057 (6)	0.3405 (11)	0.6183 (11)	0.118*

Atomic displacement parameters ($\AA^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0437 (16)	0.0508 (16)	0.0471 (16)	−0.0049 (13)	0.0018 (12)	−0.0030 (12)

C2	0.0456 (17)	0.0524 (17)	0.0471 (16)	−0.0061 (13)	−0.0019 (13)	−0.0028 (12)
C3	0.0450 (16)	0.0453 (16)	0.0429 (15)	−0.0086 (13)	0.0026 (13)	−0.0038 (12)
C4	0.0454 (17)	0.0516 (17)	0.0438 (16)	−0.0035 (14)	0.0050 (13)	0.0007 (13)
C5	0.059 (2)	0.0585 (19)	0.0576 (18)	0.0034 (15)	−0.0012 (15)	0.0016 (14)
C6	0.059 (2)	0.081 (2)	0.065 (2)	0.0065 (17)	−0.0061 (16)	0.0095 (17)
C7	0.065 (2)	0.085 (2)	0.0473 (17)	−0.0089 (19)	−0.0062 (15)	0.0023 (16)
C8	0.059 (2)	0.067 (2)	0.0457 (17)	−0.0034 (16)	−0.0011 (15)	−0.0061 (13)
C9	0.0438 (17)	0.0519 (17)	0.0410 (15)	−0.0071 (13)	0.0035 (13)	−0.0003 (13)
C10	0.0514 (18)	0.0609 (19)	0.0577 (17)	0.0003 (15)	0.0041 (14)	−0.0125 (14)
C11	0.0539 (18)	0.0449 (17)	0.0564 (18)	−0.0010 (14)	0.0019 (14)	−0.0040 (13)
C12	0.094 (3)	0.066 (2)	0.059 (2)	−0.0238 (18)	0.0002 (18)	−0.0039 (15)
C13	0.118 (3)	0.071 (2)	0.063 (2)	−0.028 (2)	0.006 (2)	−0.0001 (17)
C14	0.090 (3)	0.065 (2)	0.078 (2)	−0.0169 (19)	−0.002 (2)	−0.0156 (17)
C15	0.077 (2)	0.060 (2)	0.0617 (19)	−0.0070 (17)	0.0044 (16)	−0.0133 (15)
C16	0.0577 (19)	0.0501 (18)	0.0631 (17)	0.0000 (14)	0.0066 (14)	−0.0025 (13)
C17	0.075 (2)	0.0510 (18)	0.0643 (19)	−0.0027 (15)	0.0140 (16)	−0.0023 (14)
C18	0.0615 (19)	0.0475 (18)	0.0512 (17)	0.0001 (15)	0.0052 (15)	−0.0023 (13)
C19	0.0606 (19)	0.060 (2)	0.0489 (17)	−0.0103 (16)	0.0045 (15)	−0.0031 (14)
C20	0.081 (2)	0.087 (3)	0.060 (2)	−0.020 (2)	0.0124 (17)	−0.0076 (17)
C21	0.091 (3)	0.095 (3)	0.067 (2)	−0.033 (2)	0.0096 (19)	0.009 (2)
C22	0.097 (3)	0.065 (2)	0.091 (3)	−0.021 (2)	0.003 (2)	0.0161 (19)
C23	0.079 (2)	0.059 (2)	0.084 (2)	−0.0044 (18)	0.0083 (19)	0.0077 (17)
C24	0.0586 (19)	0.0515 (19)	0.0561 (18)	−0.0039 (15)	0.0001 (15)	0.0057 (14)
C25	0.064 (2)	0.0611 (19)	0.067 (2)	0.0088 (15)	0.0113 (17)	−0.0009 (15)
C26	0.073 (2)	0.065 (2)	0.068 (2)	−0.0030 (17)	0.0106 (18)	−0.0007 (15)
C27	0.059 (2)	0.0496 (18)	0.0570 (18)	0.0074 (15)	0.0091 (15)	−0.0030 (14)
C28	0.074 (2)	0.074 (2)	0.069 (2)	−0.0097 (17)	0.0095 (18)	0.0034 (17)
C29	0.093 (3)	0.084 (3)	0.077 (3)	−0.008 (2)	0.011 (2)	0.0129 (18)
C30	0.072 (2)	0.073 (2)	0.082 (3)	−0.0046 (18)	0.008 (2)	−0.0100 (19)
N1	0.0694 (17)	0.0542 (16)	0.0560 (15)	−0.0058 (13)	0.0115 (13)	−0.0080 (11)
N2	0.0601 (15)	0.0473 (14)	0.0556 (14)	0.0010 (12)	0.0121 (12)	0.0002 (11)
N3	0.095 (2)	0.0646 (18)	0.0815 (19)	−0.0158 (15)	0.0009 (17)	0.0012 (15)
N4	0.0495 (14)	0.0434 (13)	0.0447 (12)	−0.0027 (11)	0.0016 (11)	−0.0047 (10)
N5	0.0442 (13)	0.0499 (14)	0.0464 (13)	−0.0038 (11)	0.0001 (11)	−0.0055 (10)
N6	0.081 (2)	0.079 (2)	0.0691 (18)	0.0029 (16)	0.0082 (15)	0.0018 (15)
O1W	0.0648 (14)	0.0652 (14)	0.0756 (14)	0.0039 (11)	0.0054 (12)	−0.0102 (11)
O2W	0.0671 (15)	0.0882 (17)	0.0804 (15)	0.0068 (12)	0.0079 (13)	−0.0006 (12)

Geometric parameters (Å, °)

C1—C1[i]	1.504 (4)	C16—H16A	0.9700
C1—C2	1.520 (3)	C16—H16B	0.9700
C1—H1	0.9700	C17—C18	1.491 (3)
C1—H2	0.9700	C17—H17A	0.9700
C2—C3	1.491 (3)	C17—H17B	0.9700
C2—H3	0.9700	C18—N1	1.312 (3)
C2—H4	0.9700	C18—N2	1.369 (3)
C3—N5	1.314 (3)	C19—C20	1.384 (4)
C3—N4	1.375 (3)	C19—C24	1.390 (4)

C4—C5	1.390 (3)	C19—N1	1.391 (3)
C4—N5	1.393 (3)	C20—C21	1.375 (4)
C4—C9	1.394 (3)	C20—H20	0.9300
C5—C6	1.380 (3)	C21—C22	1.390 (4)
C5—H5	0.9300	C21—H21	0.9300
C6—C7	1.390 (4)	C22—C23	1.368 (4)
C6—H6	0.9300	C22—H22	0.9300
C7—C8	1.372 (4)	C23—C24	1.386 (3)
C7—H7	0.9300	C23—H23	0.9300
C8—C9	1.390 (3)	C24—N2	1.394 (3)
C8—H8	0.9300	C25—N2	1.467 (3)
C9—N4	1.382 (3)	C25—C27	1.503 (3)
C10—N4	1.462 (3)	C25—H25A	0.9700
C10—C11	1.516 (3)	C25—H25B	0.9700
C10—H10A	0.9700	C26—C30	1.377 (4)
C10—H10B	0.9700	C26—C27	1.383 (4)
C11—C15	1.372 (3)	C26—H26	0.9300
C11—C12	1.376 (3)	C27—C28	1.367 (3)
C12—C13	1.372 (4)	C28—C29	1.390 (4)
C12—H12	0.9300	C28—H28	0.9300
C13—N3	1.326 (3)	C29—N6	1.324 (4)
C13—H13	0.9300	C29—H29	0.9300
C14—N3	1.332 (3)	C30—N6	1.324 (4)
C14—C15	1.381 (4)	C30—H30	0.9300
C14—H14	0.9300	O1W—H1A	0.855 (16)
C15—H15	0.9300	O1W—H1B	0.830 (17)
C16—C16^{ii}	1.502 (4)	O2W—H2A	0.849 (17)
C16—C17	1.510 (3)	O2W—H2B	0.882 (16)
C1^{i}—C1—C2	112.0 (2)	C18—C17—C16	114.1 (2)
C1^{i}—C1—H1	109.2	C18—C17—H17A	108.7
C2—C1—H1	109.2	C16—C17—H17A	108.7
C1^{i}—C1—H2	109.2	C18—C17—H17B	108.7
C2—C1—H2	109.2	C16—C17—H17B	108.7
H1—C1—H2	107.9	H17A—C17—H17B	107.6
C3—C2—C1	113.7 (2)	N1—C18—N2	113.1 (2)
C3—C2—H3	108.8	N1—C18—C17	125.3 (2)
C1—C2—H3	108.8	N2—C18—C17	121.6 (2)
C3—C2—H4	108.8	C20—C19—C24	119.5 (3)
C1—C2—H4	108.8	C20—C19—N1	130.1 (3)
H3—C2—H4	107.7	C24—C19—N1	110.4 (2)
N5—C3—N4	112.3 (2)	C21—C20—C19	118.3 (3)
N5—C3—C2	125.5 (2)	C21—C20—H20	120.8
N4—C3—C2	122.2 (2)	C19—C20—H20	120.8
C5—C4—N5	129.8 (2)	C20—C21—C22	121.1 (3)
C5—C4—C9	120.1 (2)	C20—C21—H21	119.4
N5—C4—C9	110.1 (2)	C22—C21—H21	119.4
C6—C5—C4	117.6 (3)	C23—C22—C21	121.7 (3)
C6—C5—H5	121.2	C23—C22—H22	119.1

C4—C5—H5	121.2	C21—C22—H22	119.1
C5—C6—C7	121.2 (3)	C22—C23—C24	116.6 (3)
C5—C6—H6	119.4	C22—C23—H23	121.7
C7—C6—H6	119.4	C24—C23—H23	121.7
C8—C7—C6	122.4 (3)	C23—C24—C19	122.7 (3)
C8—C7—H7	118.8	C23—C24—N2	132.3 (3)
C6—C7—H7	118.8	C19—C24—N2	105.0 (2)
C7—C8—C9	116.1 (3)	N2—C25—C27	113.1 (2)
C7—C8—H8	122.0	N2—C25—H25A	109.0
C9—C8—H8	122.0	C27—C25—H25A	109.0
N4—C9—C8	132.3 (2)	N2—C25—H25B	109.0
N4—C9—C4	105.1 (2)	C27—C25—H25B	109.0
C8—C9—C4	122.6 (2)	H25A—C25—H25B	107.8
N4—C10—C11	111.8 (2)	C30—C26—C27	119.2 (3)
N4—C10—H10A	109.3	C30—C26—H26	120.4
C11—C10—H10A	109.3	C27—C26—H26	120.4
N4—C10—H10B	109.3	C28—C27—C26	117.1 (3)
C11—C10—H10B	109.3	C28—C27—C25	121.2 (3)
H10A—C10—H10B	107.9	C26—C27—C25	121.7 (3)
C15—C11—C12	116.7 (3)	C27—C28—C29	119.5 (3)
C15—C11—C10	122.1 (3)	C27—C28—H28	120.2
C12—C11—C10	121.2 (2)	C29—C28—H28	120.2
C13—C12—C11	120.6 (3)	N6—C29—C28	123.8 (3)
C13—C12—H12	119.7	N6—C29—H29	118.1
C11—C12—H12	119.7	C28—C29—H29	118.1
N3—C13—C12	123.2 (3)	N6—C30—C26	124.4 (3)
N3—C13—H13	118.4	N6—C30—H30	117.8
C12—C13—H13	118.4	C26—C30—H30	117.8
N3—C14—C15	124.1 (3)	C18—N1—C19	104.9 (2)
N3—C14—H14	118.0	C18—N2—C24	106.5 (2)
C15—C14—H14	118.0	C18—N2—C25	127.6 (2)
C11—C15—C14	119.3 (3)	C24—N2—C25	125.9 (2)
C11—C15—H15	120.4	C13—N3—C14	116.2 (3)
C14—C15—H15	120.4	C3—N4—C9	107.2 (2)
C16^{ii}—C16—C17	113.8 (3)	C3—N4—C10	128.0 (2)
C16^{ii}—C16—H16A	108.8	C9—N4—C10	124.5 (2)
C17—C16—H16A	108.8	C3—N5—C4	105.27 (19)
C16^{ii}—C16—H16B	108.8	C29—N6—C30	116.0 (3)
C17—C16—H16B	108.8	H1A—O1W—H1B	110 (2)
H16A—C16—H16B	107.7	H2A—O2W—H2B	107 (2)
C1^{i}—C1—C2—C3	178.4 (2)	N2—C25—C27—C28	116.8 (3)
C1—C2—C3—N5	−8.1 (3)	N2—C25—C27—C26	−63.9 (3)
C1—C2—C3—N4	171.3 (2)	C26—C27—C28—C29	−0.8 (4)
N5—C4—C5—C6	179.3 (2)	C25—C27—C28—C29	178.5 (3)
C9—C4—C5—C6	−1.3 (4)	C27—C28—C29—N6	−0.1 (5)
C4—C5—C6—C7	0.1 (4)	C27—C26—C30—N6	0.1 (5)
C5—C6—C7—C8	0.6 (4)	N2—C18—N1—C19	1.0 (3)
C6—C7—C8—C9	−0.1 (4)	C17—C18—N1—C19	−176.9 (2)

C7—C8—C9—N4	179.7 (2)	C20—C19—N1—C18	−179.9 (3)
C7—C8—C9—C4	−1.2 (4)	C24—C19—N1—C18	−0.2 (3)
C5—C4—C9—N4	−178.8 (2)	N1—C18—N2—C24	−1.4 (3)
N5—C4—C9—N4	0.8 (3)	C17—C18—N2—C24	176.6 (2)
C5—C4—C9—C8	1.9 (4)	N1—C18—N2—C25	176.8 (2)
N5—C4—C9—C8	−178.5 (2)	C17—C18—N2—C25	−5.2 (4)
N4—C10—C11—C15	115.2 (3)	C23—C24—N2—C18	−178.8 (3)
N4—C10—C11—C12	−63.3 (3)	C19—C24—N2—C18	1.2 (3)
C15—C11—C12—C13	0.9 (4)	C23—C24—N2—C25	3.0 (5)
C10—C11—C12—C13	179.4 (3)	C19—C24—N2—C25	−177.1 (2)
C11—C12—C13—N3	−0.5 (5)	C27—C25—N2—C18	−83.2 (3)
C12—C11—C15—C14	−0.7 (4)	C27—C25—N2—C24	94.7 (3)
C10—C11—C15—C14	−179.2 (3)	C12—C13—N3—C14	−0.1 (5)
N3—C14—C15—C11	0.1 (5)	C15—C14—N3—C13	0.3 (5)
C16^{ii}—C16—C17—C18	177.3 (3)	N5—C3—N4—C9	−0.1 (3)
C16—C17—C18—N1	4.5 (4)	C2—C3—N4—C9	−179.5 (2)
C16—C17—C18—N2	−173.3 (2)	N5—C3—N4—C10	−173.8 (2)
C24—C19—C20—C21	0.8 (4)	C2—C3—N4—C10	6.8 (4)
N1—C19—C20—C21	−179.6 (3)	C8—C9—N4—C3	178.8 (3)
C19—C20—C21—C22	0.5 (5)	C4—C9—N4—C3	−0.4 (2)
C20—C21—C22—C23	−1.7 (5)	C8—C9—N4—C10	−7.2 (4)
C21—C22—C23—C24	1.5 (5)	C4—C9—N4—C10	173.6 (2)
C22—C23—C24—C19	−0.2 (4)	C11—C10—N4—C3	98.6 (3)
C22—C23—C24—N2	179.8 (3)	C11—C10—N4—C9	−74.2 (3)
C20—C19—C24—C23	−0.9 (4)	N4—C3—N5—C4	0.5 (3)
N1—C19—C24—C23	179.4 (2)	C2—C3—N5—C4	179.9 (2)
C20—C19—C24—N2	179.1 (2)	C5—C4—N5—C3	178.7 (2)
N1—C19—C24—N2	−0.6 (3)	C9—C4—N5—C3	−0.8 (3)
C30—C26—C27—C28	0.8 (4)	C28—C29—N6—C30	1.0 (5)
C30—C26—C27—C25	−178.5 (2)	C26—C30—N6—C29	−1.0 (5)

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

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1A···N5^{iii}	0.86 (2)	2.01 (2)	2.854 (3)	171 (3)
O1W—H1B···O2W	0.83 (2)	1.97 (2)	2.800 (3)	177 (3)
O2W—H2B···N6^{iv}	0.88 (2)	2.04 (2)	2.858 (3)	154 (3)
O2W—H2A···O1W^{v}	0.85 (2)	1.93 (2)	2.779 (3)	178 (3)
C13—H13···O2W	0.93	2.53	3.390 (4)	155

Symmetry codes: (iii) −x+1, −y+1, −z+1; (iv) x, y, z+1; (v) x−1, y, z.

Fig. 1

Fig. 2

Fig. 3