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Tris(1*H*-benzimidazol-2-ylmethyl)amine methanol trisolvate

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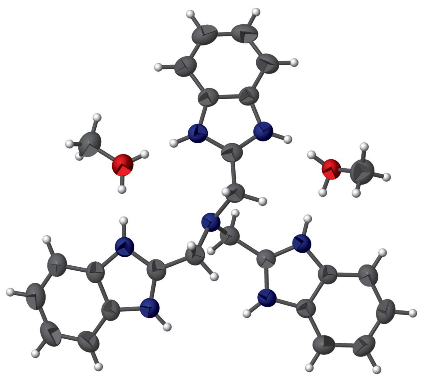
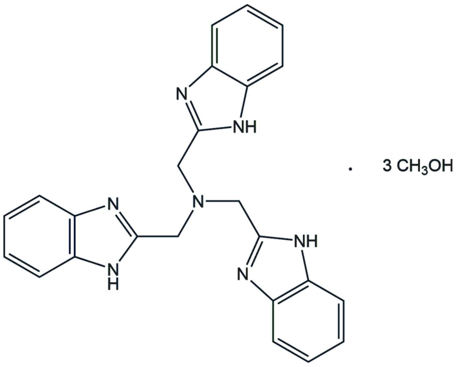
The structure of the tertiary amine tris(1*H*-benzimidazol-2-ylmethyl)amine ($C_{24}H_{21}N_7$, abbreviated ntb) has been previously reported twice as solvates, namely the monohydrate and the acetonitrile–methanol–water (1/0.5/1.5) solvate, both with the tripodal conformation formed *via* multiple hydrogen bonds. Now, we report the trimethanol adduct, ntb·3CH_3OH, where the amine has the stair conformation featuring one benzimidazole group oriented in the opposite direction from the other two. The asymmetric unit contains one-half amine, completed through the mirror plane *m* in space group $Pmn2_1$ to form the ntb molecule, with the H atom for each imidazole moiety equally disordered between both N sites available in the imidazole ring. The asymmetric unit also contains one and a half methanol molecules, one being placed in general position with the hydroxy H atom disordered over two sites with occupancy ratio 1:1, while the other lies on the *m* mirror plane, and has thus its hydroxy H atom disordered by symmetry. As in the previously reported solvates, all imine and amine groups of the ntb molecules and the methanol molecules are involved in N—H···O and O—H···N hydrogen bonds. In the title compound, however, the involved H atom is systematically a disordered H atom provided by an imidazole group or a methanol molecule.

3D view



Chemical scheme



Structure description

The tertiary amine tris(1*H*-benzimidazol-2-ylmethyl)amine (abbreviated ntb in the literature) is a molecule that can act as a tetradentate ligand through its three imine N atoms and the central amine one, exhibiting thus a versatile and rich coordination chemistry, where the molecule adopts a tripodal coordination mode to the metal (*e.g.*

Nakata *et al.*, 1997; Kwak *et al.*, 1999; Rheingold & Hammes, 2015). In addition, it is a potential hydrogen-bond donor through its three NH amine groups (Su *et al.*, 2000). Similar tripodal behaviour has been found in ntb solvent adducts such as the monohydrate, $C_{24}H_{21}N_7\cdot H_2O$, and the acetonitrile–methanol–water (1/0.5/1.5) solvate, $C_{24}H_{21}N_7\cdot C_2H_3N\cdot 0.5CH_4O\cdot 1.5H_2O$. In both cases, ntb forms two N—H⋯O and one O—H⋯N hydrogen bonds with a solvent water molecule (Zhang *et al.*, 2005). In addition, the imine N atoms of the benzimidazolyl arms can be protonated with HNO_3, and the resulting trication then adopts a stair arrangement, where the N atoms of the benzimidazolium fragments interact with anions NO_3^- through N—H⋯O hydrogen bonds (Cui, 2011).

The present report deals with a new ntb solvate. When this amine is crystallized from methanol solution, the solvate ntb·$3CH_3OH$ is obtained, in space group $Pmn2_1$. The ntb molecule has one benzimidazolyl arm placed over the mirror m plane (N2/C1⋯C5 group), while the other crystallographically independent arm (N3/N4/C6⋯C13 group) is placed in general position (Fig. 1). As a consequence, instead of the common tripodal molecular geometry (approximate point group: C_{3v}), the ntb molecule adopts a stair-like conformation (point group: C_s), with one benzimidazole group oriented in the opposite direction from the other two (Fig. 2). The imidazol H atom of the arm bisected by the mirror plane, H2, is disordered by symmetry so that the π-bond of this imidazole ring is delocalized over C2$\overset{\cdots}{-}$N2 and C2$\overset{\cdots}{-}$N2^i bonds [symmetry code: (i) $1 - x, y, z$], with a bond length of 1.342 (3) Å. The same kind of delocalization is observed in the other ntb arm, with the imidazolic H atom equally disordered over N3 and N4, and identical bond lengths C7$\overset{\cdots}{-}$N3 and C7$\overset{\cdots}{-}$N4 of 1.338 (4) and 1.335 (4) Å. In other words, amine and imine sites in ntb are indistinguishable (Fig. 2). As clearly observed in Fig. 1, the methanol molecules of crystallization are recognized by the ntb molecule in a similar way as in the previously described ionic compound ntb·$3HNO_3$ (Cui, 2011). The methanol molecules are sandwiched between two ntb arms, in order to form three O—H⋯N and three N—H⋯O hydrogen bonds (Table 1), with O⋯N separations spanning a short range, from 2.722 (4) to 2.767 (3) Å. Given that all imidazolic H atoms are disordered, the same holds for hydroxy H atoms: the first methanol molecule C14—O1 lies in the mirror m plane, with its H atom disordered by symmetry (H1); the second methanol molecule, C15—O2, placed in general position, has its hydroxy H atom disordered over two sites, H2*A* and H2*B*, with half occupancy (Fig. 2). With this arrangement, any physically unreasonable H⋯H contact is avoided, despite all heteroatoms, except N1, being involved in efficient hydrogen bonds. The complete adduct ntb·$3CH_3OH$ thus features three similar ring motifs $R_2^2(10)$ sharing the central N1 atom (Fig. 2).

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1⋯N3^i	0.84 (3)	1.95 (4)	2.762 (3)	164 (10)
O2—H2*A*⋯N2^i	0.86 (3)	1.91 (3)	2.767 (3)	174 (9)
O2—H2*B*⋯N4	0.86 (3)	1.87 (3)	2.722 (4)	171 (8)
N2—H2⋯O2^i	0.88 (3)	1.92 (3)	2.767 (3)	161 (7)
N3—H3⋯O1	0.88 (3)	1.88 (3)	2.762 (3)	176 (6)
N4—H4⋯O2	0.88 (3)	1.90 (3)	2.722 (4)	156 (3)

Symmetry code: (i) $-x + 1, y, z$.

The supramolecular structure is further extended by one-dimensional stacks of ntb molecules in the [011] direction. The stair conformation adopted by ntb allows rather close π–π interactions between benzimidazole rings along the stacks: mean plane separations for neighbouring rings along a stack are 3.782 (2) and 3.454 (2) Å for the N2/C2–C5 and N3/N4/C7–C13 benzimidazole rings, respectively.

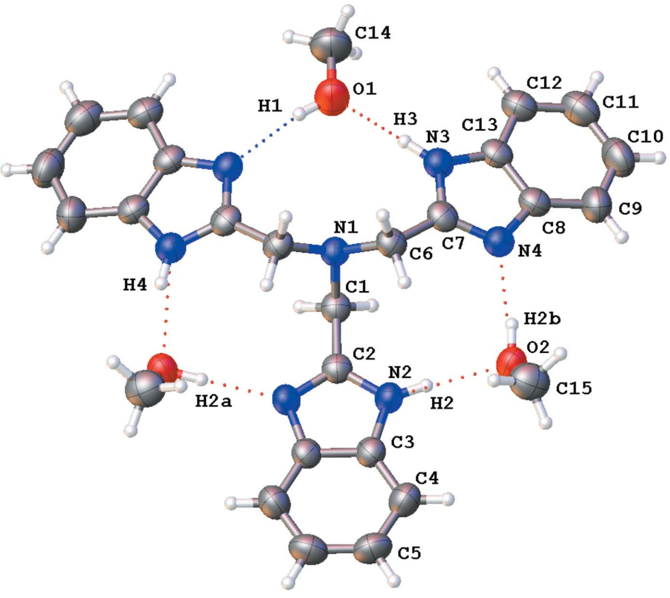


Figure 1
Molecular structure of the title compound, with displacement ellipsoids for non-H atoms at the 50% probability level. For all disordered H atoms, a single site was retained, in order to emphasize the O—H⋯N and N—H⋯O hydrogen bonds (dotted lines). Non-labelled atoms are generated by the symmetry operation $1 - x, y, z$.

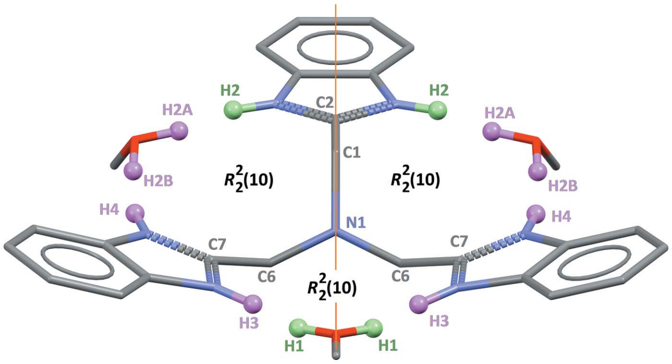


Figure 2
View of the title compound rotated by *ca* 90° in comparison with Fig. 1, showing the stair-like conformation for ntb. All H atoms bonded to heteroatoms are shown. Green H atoms are disordered by symmetry, while purple H atoms are positionally disordered, all having the same half occupancy. The orange line is the trace of the m mirror normal to the a axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{21}N_7 \cdot 3CH_4O$
M_r	503.60
Crystal system, space group	Orthorhombic, $Pmn2_1$
Temperature (K)	293
a, b, c (Å)	16.7781 (17), 16.3902 (18), 4.7894 (5)
V (Å^3)	1317.1 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.77 × 0.17 × 0.10
Data collection	
Diffractometer	Agilent Xcalibur, Atlas, Gemini
Absorption correction	Analytical (*CrysAlis PRO*; Agilent, 2012)
$T_{\min}$, $T_{\max}$	0.975, 0.995
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8328, 2938, 1903
$R_{\rm int}$	0.047
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.101, 1.03
No. of reflections	2938
No. of parameters	196
No. of restraints	11
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.14, −0.14

Computer programs: *CrysAlis PRO* and *CrysAlis RED* (Agilent, 2012), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

The title compound was prepared through the solid-solid condensation reaction of nitrilotriacetic acid (1 g, 5.2 mmol) and *o*-phenylenediamine (1.7 g, 15.7 mmol), both reactants contained in a round-bottom flask provided with a septum connected to the outside through a needle, which was maintained at 463–473 K in a sand bath for 1 h. As the heating progressed, a dark-brown solution formed, which solidified when the temperature returned to 298 K. Then, 0.7 g of activated carbon and 20 ml of methanol were added and the mixture refluxed for 2 h. The still hot mixture was filtered in a Büchner funnel, giving an orange filtrate. The product was isolated by successive recrystallizations from methanol in 30% yield, and single crystals grew by slow evaporation at 298 K as colourless needles with a melting point of 543–547 K. IR (KBr): 3391, 3177, 1624, 1535, 1437, 1273, 1119, 737 cm^{-1}.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal emulates a tetragonal system ($a \simeq b$), however, extinctions and structure refinement are consistent with space group $Pmn2_1$. The imidazole ring in a general position (N3/H3/N4/H4/C7/C8/C13) was restrained to be flat, within a standard deviation of 0.1 Å^3 (Sheldrick, 2015*b*).

Acknowledgements

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full crystallographic data

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Tris(1*H*-benzimidazol-2-ylmethyl)amine methanol trisolvate

Crystal data

$C_{24}H_{21}N_7 \cdot 3CH_4O$
$M_r = 503.60$
Orthorhombic, $Pmn2_1$
$a = 16.7781$ (17) Å
$b = 16.3902$ (18) Å
$c = 4.7894$ (5) Å
$V = 1317.1$ (2) Å^3
$Z = 2$
$F(000) = 536$

$D_x = 1.270$ Mg m^{-3}
Melting point: 543 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1461 reflections
$\theta = 3.8$–$23.1°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Needle, colourless
0.77 × 0.17 × 0.10 mm

Data collection

Agilent Xcalibur, Atlas, Gemini diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.5564 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlis PRO; Agilent, 2012)

$T_{\min} = 0.975$, $T_{\max} = 0.995$
8328 measured reflections
2938 independent reflections
1903 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.047$
$\theta_{\max} = 27.1°$, $\theta_{\min} = 3.5°$
$h = -21 \rightarrow 21$
$k = -20 \rightarrow 20$
$l = -6 \rightarrow 6$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.101$
$S = 1.03$
2938 reflections
196 parameters
11 restraints
0 constraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (2)

Special details

Refinement. All C-bonded H atoms were placed in calculated positions and refined as riding atoms, while H atoms bonded to N (H2, H3, H4) and O sites (H1, H2A, H2B) were found in difference maps, and refined with free coordinates and occupancy fixed to 1/2. Corresponding bond lengths were restrained to O—H = 0.85 (2) and N—H = 0.90 (2) Å.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.500000	0.7530 (2)	0.4852 (7)	0.0411 (8)	
N2	0.43371 (14)	0.56374 (16)	0.6509 (6)	0.0483 (6)	
H2	0.3840 (19)	0.578 (4)	0.616 (15)	0.058*	0.5
N3	0.63616 (15)	0.85800 (15)	0.2727 (6)	0.0481 (7)	
H3	0.593 (3)	0.886 (3)	0.231 (13)	0.058*	0.5
N4	0.71198 (16)	0.76093 (15)	0.4609 (6)	0.0517 (7)	
H4	0.725 (4)	0.7189 (17)	0.565 (8)	0.062*	0.5
C1	0.500000	0.6713 (3)	0.3595 (8)	0.0456 (11)	
H1A	0.546531	0.667019	0.240219	0.055*	0.5
H1B	0.453469	0.667019	0.240219	0.055*	0.5
C2	0.500000	0.5995 (3)	0.5526 (8)	0.0441 (11)	
C3	0.45864 (17)	0.5001 (2)	0.8202 (7)	0.0467 (8)	
C4	0.4157 (2)	0.4429 (2)	0.9728 (8)	0.0620 (9)	
H4A	0.360238	0.442753	0.973673	0.074*	
C5	0.4586 (2)	0.3865 (2)	1.1222 (9)	0.0674 (10)	
H5	0.431646	0.347357	1.226065	0.081*	
C6	0.57325 (16)	0.77133 (19)	0.6402 (7)	0.0461 (7)	
H6A	0.588978	0.723567	0.746291	0.055*	
H6B	0.562722	0.815084	0.771494	0.055*	
C7	0.64012 (19)	0.79584 (19)	0.4532 (7)	0.0471 (8)	
C8	0.75915 (19)	0.80341 (19)	0.2720 (7)	0.0495 (8)	
C9	0.8387 (2)	0.7945 (2)	0.1972 (8)	0.0657 (10)	
H9	0.870759	0.754618	0.277911	0.079*	
C10	0.8681 (2)	0.8473 (3)	−0.0016 (9)	0.0733 (11)	
H10	0.920992	0.842674	−0.057055	0.088*	
C11	0.8205 (3)	0.9071 (2)	−0.1211 (9)	0.0712 (11)	
H11	0.842493	0.941876	−0.253804	0.085*	
C12	0.7416 (2)	0.9164 (2)	−0.0482 (8)	0.0590 (9)	
H12	0.709832	0.956296	−0.129831	0.071*	
C13	0.71147 (18)	0.86342 (18)	0.1529 (7)	0.0470 (8)	
C14	0.500000	1.0001 (4)	−0.0870 (13)	0.0875 (19)	
H14A	0.477295	1.049629	−0.015783	0.131*	0.5
H14B	0.468974	0.981180	−0.242542	0.131*	0.5
H14C	0.553731	1.010105	−0.146720	0.131*	0.5
O1	0.500000	0.9415 (3)	0.1199 (10)	0.0815 (11)	
H1	0.458 (3)	0.923 (6)	0.19 (2)	0.122*	0.5
C15	0.7479 (2)	0.5979 (2)	0.9575 (8)	0.0747 (11)	
H15A	0.768010	0.543691	0.985412	0.112*	
H15B	0.702487	0.606585	1.075551	0.112*	
H15C	0.788684	0.636824	1.003105	0.112*	
O2	0.72533 (13)	0.60776 (15)	0.6755 (6)	0.0605 (7)	
H2A	0.676 (2)	0.595 (5)	0.68 (2)	0.091*	0.5
H2B	0.727 (5)	0.657 (2)	0.611 (17)	0.091*	0.5

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.043 (2)	0.043 (2)	0.0379 (19)	0.000	0.000	−0.0032 (16)
N2	0.0407 (14)	0.0496 (16)	0.0546 (14)	−0.0032 (13)	−0.0037 (15)	0.0040 (14)
N3	0.0464 (16)	0.0483 (16)	0.0496 (15)	−0.0033 (12)	−0.0017 (14)	0.0020 (15)
N4	0.0483 (16)	0.0488 (16)	0.0580 (16)	−0.0015 (14)	−0.0008 (15)	0.0056 (14)
C1	0.046 (2)	0.050 (3)	0.041 (2)	0.000	0.000	−0.004 (2)
C2	0.047 (3)	0.044 (3)	0.041 (3)	0.000	0.000	−0.0046 (19)
C3	0.0454 (17)	0.0455 (18)	0.0492 (16)	−0.0028 (14)	−0.0037 (15)	−0.0035 (15)
C4	0.052 (2)	0.062 (2)	0.072 (2)	−0.0085 (18)	0.001 (2)	0.006 (2)
C5	0.078 (2)	0.053 (2)	0.072 (2)	−0.0092 (17)	0.004 (2)	0.013 (2)
C6	0.0480 (18)	0.0465 (18)	0.0436 (16)	−0.0053 (14)	−0.0038 (17)	−0.0014 (16)
C7	0.050 (2)	0.0470 (19)	0.0445 (17)	−0.0089 (16)	−0.0031 (16)	−0.0069 (17)
C8	0.052 (2)	0.0430 (18)	0.0535 (19)	−0.0070 (16)	−0.0004 (17)	−0.0004 (17)
C9	0.048 (2)	0.067 (2)	0.082 (3)	−0.0003 (18)	0.006 (2)	0.001 (2)
C10	0.056 (2)	0.079 (3)	0.085 (3)	−0.013 (2)	0.017 (2)	−0.003 (2)
C11	0.076 (3)	0.065 (3)	0.073 (3)	−0.016 (2)	0.013 (2)	0.002 (2)
C12	0.065 (2)	0.051 (2)	0.061 (2)	−0.0083 (17)	0.003 (2)	0.0023 (19)
C13	0.0523 (19)	0.0416 (18)	0.0472 (17)	−0.0087 (15)	−0.0005 (18)	−0.0047 (16)
C14	0.088 (4)	0.076 (4)	0.099 (5)	0.000	0.000	0.030 (4)
O1	0.061 (2)	0.085 (3)	0.099 (3)	0.000	0.000	0.039 (2)
C15	0.084 (3)	0.072 (3)	0.068 (3)	−0.006 (2)	−0.009 (2)	0.004 (2)
O2	0.0478 (13)	0.0639 (16)	0.0698 (17)	0.0046 (13)	0.0019 (15)	0.0169 (14)

Geometric parameters (Å, º)

N1—C6^{i}	1.467 (3)	C6—H6B	0.9700
N1—C6	1.467 (3)	C8—C9	1.390 (4)
N1—C1	1.468 (5)	C8—C13	1.390 (4)
N2—C2	1.342 (3)	C9—C10	1.377 (5)
N2—C3	1.386 (4)	C9—H9	0.9300
N2—H2	0.88 (3)	C10—C11	1.388 (5)
N3—C7	1.338 (4)	C10—H10	0.9300
N3—C13	1.391 (4)	C11—C12	1.378 (5)
N3—H3	0.88 (3)	C11—H11	0.9300
N4—C7	1.335 (4)	C12—C13	1.391 (4)
N4—C8	1.389 (4)	C12—H12	0.9300
N4—H4	0.88 (3)	C14—O1	1.379 (6)
C1—C2	1.496 (6)	C14—H14A	0.9600
C1—H1A	0.9700	C14—H14B	0.9600
C1—H1B	0.9700	C14—H14C	0.9600
C3—C3^{i}	1.388 (6)	O1—H1	0.84 (3)
C3—C4	1.391 (4)	O1—H1^{i}	0.84 (3)
C4—C5	1.373 (5)	C15—O2	1.412 (4)
C4—H4A	0.9300	C15—H15A	0.9600
C5—C5^{i}	1.388 (7)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600

C6—C7	1.491 (4)	O2—H2A	0.86 (3)
C6—H6A	0.9700	O2—H2B	0.86 (3)

C6^{i}—N1—C6	113.8 (3)	N3—C7—C6	123.8 (3)
C6^{i}—N1—C1	113.3 (2)	N4—C8—C9	131.5 (3)
C6—N1—C1	113.3 (2)	N4—C8—C13	107.1 (3)
C2—N2—C3	106.5 (3)	C9—C8—C13	121.4 (3)
C2—N2—H2	127 (4)	C10—C9—C8	117.1 (4)
C3—N2—H2	127 (4)	C10—C9—H9	121.5
C7—N3—C13	105.7 (3)	C8—C9—H9	121.5
C7—N3—H3	126 (4)	C9—C10—C11	121.6 (4)
C13—N3—H3	129 (4)	C9—C10—H10	119.2
C7—N4—C8	106.4 (3)	C11—C10—H10	119.2
C7—N4—H4	125 (4)	C12—C11—C10	121.7 (4)
C8—N4—H4	128 (4)	C12—C11—H11	119.1
N1—C1—C2	117.6 (3)	C10—C11—H11	119.1
N1—C1—H1A	107.9	C11—C12—C13	117.1 (3)
C2—C1—H1A	107.9	C11—C12—H12	121.4
N1—C1—H1B	107.9	C13—C12—H12	121.4
C2—C1—H1B	107.9	C8—C13—N3	108.0 (3)
H1A—C1—H1B	107.2	C8—C13—C12	121.1 (3)
N2—C2—N2^{i}	111.9 (4)	N3—C13—C12	130.9 (3)
N2—C2—C1	124.06 (19)	O1—C14—H14A	109.5
N2^{i}—C2—C1	124.06 (19)	O1—C14—H14B	109.5
N2—C3—C3^{i}	107.56 (15)	H14A—C14—H14B	109.5
N2—C3—C4	131.2 (3)	O1—C14—H14C	109.5
C3^{i}—C3—C4	121.2 (2)	H14A—C14—H14C	109.5
C5—C4—C3	117.1 (3)	H14B—C14—H14C	109.5
C5—C4—H4A	121.5	C14—O1—H1	122 (7)
C3—C4—H4A	121.5	C14—O1—H1^{i}	122 (7)
C4—C5—C5^{i}	121.7 (2)	H1—O1—H1^{i}	116 (10)
C4—C5—H5	119.2	O2—C15—H15A	109.5
C5^{i}—C5—H5	119.2	O2—C15—H15B	109.5
N1—C6—C7	112.4 (3)	H15A—C15—H15B	109.5
N1—C6—H6A	109.1	O2—C15—H15C	109.5
C7—C6—H6A	109.1	H15A—C15—H15C	109.5
N1—C6—H6B	109.1	H15B—C15—H15C	109.5
C7—C6—H6B	109.1	C15—O2—H2A	102 (7)
H6A—C6—H6B	107.8	C15—O2—H2B	116 (6)
N4—C7—N3	112.9 (3)	H2A—O2—H2B	106 (7)
N4—C7—C6	123.2 (3)		

C6^{i}—N1—C1—C2	−65.7 (2)	N1—C6—C7—N4	−127.8 (3)
C6—N1—C1—C2	65.8 (2)	N1—C6—C7—N3	56.5 (4)
C3—N2—C2—N2^{i}	−0.9 (5)	C7—N4—C8—C9	179.1 (4)
C3—N2—C2—C1	179.8 (3)	C7—N4—C8—C13	−0.7 (3)
N1—C1—C2—N2	89.6 (4)	N4—C8—C9—C10	179.8 (3)
N1—C1—C2—N2^{i}	−89.6 (4)	C13—C8—C9—C10	−0.5 (5)

C2—N2—C3—C3^{i}	0.5 (3)	C8—C9—C10—C11	0.4 (6)
C2—N2—C3—C4	−179.5 (3)	C9—C10—C11—C12	−0.4 (6)
N2—C3—C4—C5	179.9 (4)	C10—C11—C12—C13	0.5 (6)
C3^{i}—C3—C4—C5	−0.2 (4)	N4—C8—C13—N3	0.9 (3)
C3—C4—C5—C5^{i}	0.2 (4)	C9—C8—C13—N3	−178.9 (3)
C6^{i}—N1—C6—C7	−149.0 (2)	N4—C8—C13—C12	−179.6 (3)
C1—N1—C6—C7	79.7 (4)	C9—C8—C13—C12	0.6 (5)
C8—N4—C7—N3	0.2 (4)	C7—N3—C13—C8	−0.8 (3)
C8—N4—C7—C6	−176.0 (3)	C7—N3—C13—C12	179.8 (3)
C13—N3—C7—N4	0.4 (3)	C11—C12—C13—C8	−0.6 (5)
C13—N3—C7—C6	176.5 (3)	C11—C12—C13—N3	178.8 (3)

Symmetry code: (i) −*x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3^{i}	0.84 (3)	1.95 (4)	2.762 (3)	164 (10)
O2—H2*A*···N2^{i}	0.86 (3)	1.91 (3)	2.767 (3)	174 (9)
O2—H2*B*···N4	0.86 (3)	1.87 (3)	2.722 (4)	171 (8)
N2—H2···O2^{i}	0.88 (3)	1.92 (3)	2.767 (3)	161 (7)
N3—H3···O1	0.88 (3)	1.88 (3)	2.762 (3)	176 (6)
N4—H4···O2	0.88 (3)	1.90 (3)	2.722 (4)	156 (3)

Symmetry code: (i) −*x*+1, *y*, *z*.