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Crystal structure of 1-methylimidazole 3-oxide monohydrate

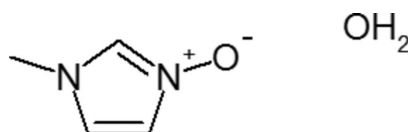
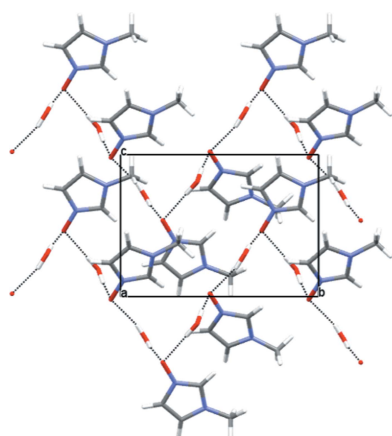
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1-Methylimidazole 3-*N*-oxide (NMI-O) crystallizes as a monohydrate, C₄H₆N₂O·H₂O, in the monoclinic space group *P*2₁ with *Z*' = 2 (molecules *A* and *B*). The imidazole rings display a planar geometry (r.m.s. deviations = 0.0008 and 0.0002 Å) and are linked in the crystal structure into infinite zigzag strands of ···NMI-O(*A*)···OH₂···NMI-O(*B*)···OH₂··· units by O—H···O hydrogen bonds. These chains propagate along the *b*-axis direction of the unit cell.

1. Chemical context

Aryl-*N*-oxides are an important class of materials acting as highly efficient catalysts for the phosphorylation of alcohols (Murray *et al.*, 2015) and also for the site-selective phosphorylation of polyols and peptides (Murray *et al.* 2014). One material in particular, 1-methylimidazole 3-*N*-oxide, (NMI-O), has been shown to be a highly efficient catalyst for both sulfonylation and silylation procedures (Murray & Spivey, 2015). Until recently, NMI-O has been somewhat elusive in the literature. The synthesis of NMI-O and its use as a highly efficient catalyst for certain Morita–Baylis–Hillman reactions has been reported (Lin *et al.*, 2005) although no conclusive information on the structural identity of the material synthesized was presented. A recent paper, directed at the synthesis of salts of 1-alkyl-imidazole 3-oxides for use as ionic liquids also reported the synthesis of NMI-O, however all attempts at crystallizing a sample of this material were unsuccessful although two crystalline adducts of NMI-O, a tris (2-thienyl)-borane and a silver carbene hexafluoridophosphate, were structurally characterized (Laus *et al.*, 2008). These authors also demonstrated by NMR and subsequent X-ray structural analysis of a related 1,2-dimethylimidazole semiperhydrate material that the likely product reported earlier (Lin *et al.*, 2005) was the 1-methylimidazole semiperhydrate rather than NMI-O itself. We now present a simplified synthesis of NMI-O and the crystal structure of its hydrate.



2. Structural commentary

The asymmetric unit of the title compound is shown in Fig. 1. It contains two molecules of NMI-O and two fully occupied

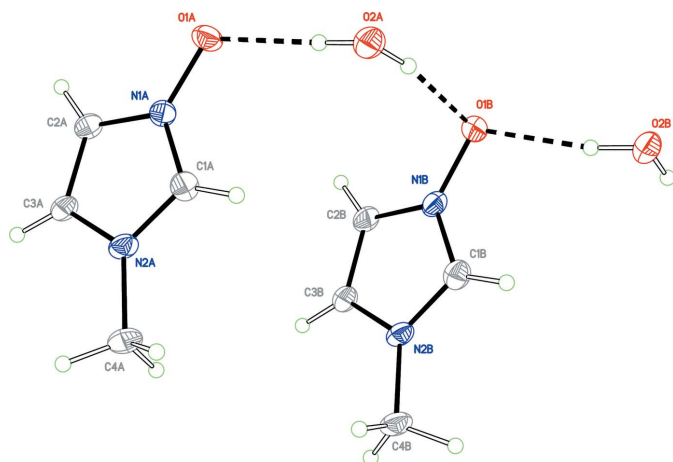


Figure 1
View of the asymmetric unit of the title compound with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The O—H...O hydrogen bonds are shown as dashed lines.

and ordered water molecules, making the overall stoichiometry a monohydrate. A calculated least-squares plane through the five atoms of the imidazole ring (C1, N1, C2, C3, N2) for molecules *A* and *B* gave r.m.s. deviations from planarity of 0.0008 and 0.0002 Å, respectively, with the oxygen atoms of the N⁺—O[−] groups also residing close to the ring plane; O1A, −0.021 (4) Å; O1B, −0.008 (4) Å. The methyl groups lie somewhat farther outside the plane of the ring with displacements of −0.073 (5) Å for C4A and −0.116 (1) Å for

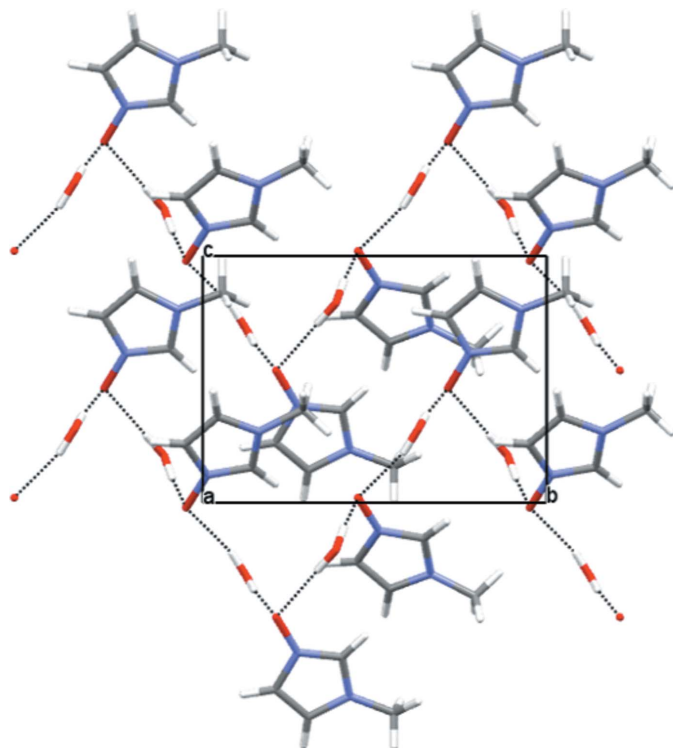


Figure 2
View of the crystal packing down the *a* axis. The O—H...O hydrogen bonds (see Table 1) are shown as dotted lines.

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2A—H2AA...O1B	1.03 (6)	1.73 (6)	2.752 (3)	172 (4)
O2A—H2AB...O1A	0.83 (5)	1.94 (5)	2.773 (3)	175 (4)
O2B—H2BA...O1B	0.83 (4)	1.94 (4)	2.752 (3)	167 (4)
O2B—H2BB...O1A ⁱ	0.94 (5)	1.86 (5)	2.790 (3)	171 (5)
C1A—H1A...O1A ⁱⁱ	0.95	2.47	3.248 (4)	139
C4A—H4AC...O1A ⁱⁱ	0.98	2.46	3.308 (4)	145
C4B—H4BC...O1A ⁱⁱ	0.98	2.56	3.336 (4)	136
C1B—H1B...O1B ⁱ	0.95	2.48	3.248 (4)	138
C2B—H2B...O2B ⁱⁱⁱ	0.95	2.41	3.298 (4)	155
C4B—H4BA...O1B ⁱ	0.98	2.50	3.345 (4)	144

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

C4B. The dihedral angle formed between the least-squares planes of the *A* and *B* NMI-O molecules is 12.96 (16)°. The present data were not of sufficient quality to determine the absolute structure.

3. Supramolecular features

In the crystal, the NMI-O and water molecules are linked by O—H...O hydrogen bonds to form an infinite NMI-O...OH₂...NMI-O...OH₂... chain propagating along the *b*-axis direction of the unit cell. Each water molecule forms two hydrogen bonds, one to each of the N⁺—O[−] groups of NMI-O molecules *A* and *B* with the oxygen atoms of these groups acting as double acceptors from both water molecules (Table 1, Fig. 2). The NMI-O...OH₂...NMI-O...OH₂... chains are cross-linked in the crystal structure by weaker C—H...O interactions (Table 1) with H...O contacts in the range 2.41–2.56 Å.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.37 update February 2016; Groom *et al.*, 2016) for the imidazole-3-oxide substructure yielded 16 hits, all of which were genuine examples of substituted imidazole-3-oxides. Closely related examples include 1-hydroxyimidazole-3-oxide (DOJKUJ), 1-hydroxy-2-methylimidazole-3-oxide (DOJLAQ), 3-hydroxy-1,2-dimethylimidazolium 1,2-dimethylimidazolium-3-oxide iodide (DOJMUL) and 1,2-dimethylimidazole-3-oxide (DOJNAS) (Laus *et al.*, 2008). For 1-hydroxy-2,4,5-triphenyl-1*H*-imidazole 3-oxide (JADNAE; Sánchez-Migallón *et al.* 2003), the N⁺—O[−] bond length was particularly short at 1.276 and 1.278 Å for the two molecules in the asymmetric unit. For the title compound, the N⁺—O[−] bond lengths are 1.350 (3) and 1.348 (3) Å for molecules *A* and *B*, respectively. These values are within the range exhibited for the remaining 15 database entries (1.326–1.368 Å).

5. Synthesis and crystallization

The title compound was synthesized in a three-step, one-pot process in which aqueous glyoxal was condensed with

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₄ H ₆ N ₂ O·H ₂ O
<i>M_r</i>	116.12
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.5941 (6), 10.0703 (6), 7.8286 (6)
β (°)	112.402 (9)
<i>V</i> (Å ³)	553.51 (8)
<i>Z</i>	4
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	0.95
Crystal size (mm)	0.45 × 0.10 × 0.05
Data collection	
Diffraction	Rigaku SuperNova, Dualflex, AtlasS2
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.419, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	2067, 1386, 1241
<i>R_{int}</i>	0.023
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.624
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.042, 0.119, 1.01
No. of reflections	1386
No. of parameters	163
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.21, -0.23

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXD2014* (Sheldrick *et al.*, 2001), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *pubCIF* (Westrip, 2010).

hydroxylamine hydrochloride in the presence of sodium carbonate to afford the mono-oxime. This intermediate was immediately condensed with methylamine to give the corresponding imine, which cyclo-condenses upon exposure to aqueous formaldehyde to give NMI-O after acidic workup in ~68% yield (Murray & Spivey, 2016). The previously reported synthesis also started from glyoxal but required eight steps (Laus *et al.*, 2008). The material was concentrated *in vacuo* to afford a brown oil, which crystallized overnight as colourless laths in the freezer after exposure to air, forming a mono-hydrate species. The crystals as prepared were extremely

hygroscopic, necessitating a rapid transfer to the cold stream of the diffractometer.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The four water H atoms were located in a Fourier difference map and freely refined. All the remaining H atoms were placed geometrically in idealized positions and allowed to ride on their parent atoms: C–H = 0.95–0.98 Å with *U*_{iso}(H) = 1.5*U*_{eq}(C-methyl) and *U*_{iso}(H) = 1.2*U*_{eq}(C) for other H atoms. The data were not of a sufficient quality to reliably determine the absolute structure.

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: *SHELXD2014* (Sheldrick *et al.*, 2001); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

1-Methylimidazole 3-*N*-oxide monohydrate

Crystal data

$C_4H_6N_2O \cdot H_2O$

$M_r = 116.12$

Monoclinic, $P2_1$

$a = 7.5941$ (6) Å

$b = 10.0703$ (6) Å

$c = 7.8286$ (6) Å

$\beta = 112.402$ (9)°

$V = 553.51$ (8) Å³

$Z = 4$

$F(000) = 248$

$D_x = 1.393$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 1007 reflections

$\theta = 6.3$ – 74.8°

$\mu = 0.95$ mm⁻¹

$T = 100$ K

Lath, colourless

$0.45 \times 0.10 \times 0.05$ mm

Data collection

Rigaku SuperNova, Dualflex, AtlasS2
diffractometer

Radiation source: fine-focus sealed X-ray tube,
Enhance (Cu) X-ray Source

Detector resolution: 5.2921 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*CrysAlis PRO*; Rigaku OD, 2015)

$T_{\min} = 0.419$, $T_{\max} = 1.000$

2067 measured reflections

1386 independent reflections

1241 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 74.3^\circ$, $\theta_{\min} = 6.1^\circ$

$h = -9 \rightarrow 8$

$k = -12 \rightarrow 5$

$l = -9 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.119$

$S = 1.01$

1386 reflections

163 parameters

1 restraint

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.21$ e Å⁻³

$\Delta\rho_{\min} = -0.23$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1A	0.5193 (3)	0.2133 (2)	0.5360 (3)	0.0218 (5)
N1A	0.3842 (3)	0.2757 (3)	0.3934 (3)	0.0176 (6)
N2A	0.2057 (4)	0.4269 (3)	0.2161 (4)	0.0189 (6)
C1A	0.3553 (4)	0.4046 (3)	0.3747 (4)	0.0193 (6)
H1A	0.4276	0.4707	0.4589	0.023*
C2A	0.2517 (4)	0.2107 (3)	0.2439 (4)	0.0185 (6)
H2A	0.2413	0.1176	0.2231	0.022*
C3A	0.1392 (4)	0.3066 (3)	0.1325 (4)	0.0185 (6)
H3A	0.0348	0.2931	0.0187	0.022*
C4A	0.1200 (4)	0.5563 (3)	0.1484 (5)	0.0231 (7)
H4AA	0.0184	0.5739	0.1936	0.035*
H4AB	0.0667	0.5562	0.0130	0.035*
H4AC	0.2176	0.6256	0.1934	0.035*
O2A	0.7412 (3)	0.3868 (2)	0.8081 (3)	0.0237 (5)
H2AA	0.675 (7)	0.410 (6)	0.898 (7)	0.063 (16)*
H2AB	0.669 (6)	0.335 (5)	0.729 (6)	0.039 (13)*
O1B	0.5360 (3)	0.4513 (2)	1.0200 (3)	0.0226 (5)
N1B	0.3895 (4)	0.5126 (3)	0.8873 (3)	0.0188 (6)
N2B	0.2080 (4)	0.6632 (3)	0.7115 (4)	0.0183 (6)
C1B	0.3733 (4)	0.6430 (3)	0.8560 (4)	0.0203 (7)
H1B	0.4617	0.7092	0.9231	0.024*
C2B	0.2336 (4)	0.4478 (3)	0.7620 (4)	0.0201 (6)
H2B	0.2105	0.3548	0.7544	0.024*
C3B	0.1191 (4)	0.5435 (3)	0.6509 (4)	0.0194 (6)
H3B	0.0007	0.5300	0.5510	0.023*
C4B	0.1431 (4)	0.7915 (3)	0.6217 (5)	0.0222 (7)
H4BA	0.1842	0.8624	0.7144	0.033*
H4BB	0.0038	0.7916	0.5622	0.033*
H4BC	0.1978	0.8065	0.5284	0.033*
O2B	0.7272 (3)	0.6268 (2)	1.2987 (3)	0.0242 (6)
H2BA	0.663 (6)	0.585 (5)	1.205 (5)	0.023 (10)*
H2BB	0.638 (7)	0.648 (6)	1.351 (7)	0.057 (15)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0226 (10)	0.0189 (12)	0.0202 (12)	0.0033 (9)	0.0039 (10)	0.0048 (10)
N1A	0.0212 (12)	0.0148 (13)	0.0171 (12)	0.0005 (9)	0.0075 (10)	0.0015 (10)
N2A	0.0236 (12)	0.0108 (14)	0.0229 (13)	0.0007 (10)	0.0095 (11)	0.0008 (10)

C1A	0.0210 (13)	0.0169 (16)	0.0196 (15)	-0.0029 (12)	0.0071 (12)	-0.0001 (11)
C2A	0.0228 (14)	0.0103 (15)	0.0223 (15)	-0.0013 (12)	0.0084 (13)	-0.0007 (12)
C3A	0.0227 (14)	0.0101 (14)	0.0219 (15)	-0.0012 (11)	0.0077 (12)	-0.0025 (12)
C4A	0.0290 (15)	0.0107 (15)	0.0300 (16)	0.0013 (12)	0.0117 (14)	0.0027 (13)
O2A	0.0248 (11)	0.0177 (13)	0.0263 (12)	-0.0010 (9)	0.0071 (10)	-0.0023 (10)
O1B	0.0256 (11)	0.0192 (11)	0.0185 (10)	0.0039 (9)	0.0032 (9)	-0.0010 (9)
N1B	0.0243 (13)	0.0137 (14)	0.0187 (13)	0.0015 (10)	0.0086 (11)	-0.0017 (9)
N2B	0.0229 (12)	0.0110 (13)	0.0220 (12)	0.0005 (10)	0.0097 (10)	-0.0001 (10)
C1B	0.0230 (15)	0.0181 (16)	0.0209 (14)	-0.0025 (12)	0.0097 (13)	-0.0020 (12)
C2B	0.0263 (15)	0.0111 (14)	0.0224 (14)	-0.0015 (12)	0.0087 (12)	-0.0012 (11)
C3B	0.0217 (13)	0.0139 (15)	0.0210 (13)	-0.0024 (12)	0.0063 (12)	-0.0032 (12)
C4B	0.0280 (15)	0.0108 (15)	0.0279 (16)	0.0015 (13)	0.0107 (14)	0.0025 (12)
O2B	0.0265 (12)	0.0200 (14)	0.0251 (11)	-0.0013 (10)	0.0086 (10)	-0.0053 (10)

Geometric parameters (Å, °)

O1A—N1A	1.350 (3)	O1B—N1B	1.348 (3)
N1A—C1A	1.315 (4)	N1B—C1B	1.332 (4)
N1A—C2A	1.384 (4)	N1B—C2B	1.380 (4)
N2A—C1A	1.344 (4)	N2B—C1B	1.348 (4)
N2A—C3A	1.378 (4)	N2B—C3B	1.374 (4)
N2A—C4A	1.463 (4)	N2B—C4B	1.464 (4)
C1A—H1A	0.9500	C1B—H1B	0.9500
C2A—C3A	1.362 (4)	C2B—C3B	1.366 (4)
C2A—H2A	0.9500	C2B—H2B	0.9500
C3A—H3A	0.9500	C3B—H3B	0.9500
C4A—H4AA	0.9800	C4B—H4BA	0.9800
C4A—H4AB	0.9800	C4B—H4BB	0.9800
C4A—H4AC	0.9800	C4B—H4BC	0.9800
O2A—H2AA	1.03 (6)	O2B—H2BA	0.83 (4)
O2A—H2AB	0.83 (5)	O2B—H2BB	0.94 (5)
C1A—N1A—O1A	126.5 (3)	C1B—N1B—O1B	125.7 (3)
C1A—N1A—C2A	109.6 (3)	C1B—N1B—C2B	110.0 (3)
O1A—N1A—C2A	123.9 (3)	O1B—N1B—C2B	124.3 (3)
C1A—N2A—C3A	108.6 (3)	C1B—N2B—C3B	109.6 (3)
C1A—N2A—C4A	125.8 (3)	C1B—N2B—C4B	124.9 (3)
C3A—N2A—C4A	125.4 (3)	C3B—N2B—C4B	125.3 (3)
N1A—C1A—N2A	108.3 (3)	N1B—C1B—N2B	107.1 (3)
N1A—C1A—H1A	125.8	N1B—C1B—H1B	126.4
N2A—C1A—H1A	125.8	N2B—C1B—H1B	126.4
C3A—C2A—N1A	106.4 (3)	C3B—C2B—N1B	106.5 (3)
C3A—C2A—H2A	126.8	C3B—C2B—H2B	126.7
N1A—C2A—H2A	126.8	N1B—C2B—H2B	126.7
C2A—C3A—N2A	107.0 (3)	C2B—C3B—N2B	106.8 (3)
C2A—C3A—H3A	126.5	C2B—C3B—H3B	126.6
N2A—C3A—H3A	126.5	N2B—C3B—H3B	126.6
N2A—C4A—H4AA	109.5	N2B—C4B—H4BA	109.5

N2A—C4A—H4AB	109.5	N2B—C4B—H4BB	109.5
H4AA—C4A—H4AB	109.5	H4BA—C4B—H4BB	109.5
N2A—C4A—H4AC	109.5	N2B—C4B—H4BC	109.5
H4AA—C4A—H4AC	109.5	H4BA—C4B—H4BC	109.5
H4AB—C4A—H4AC	109.5	H4BB—C4B—H4BC	109.5
H2AA—O2A—H2AB	107 (4)	H2BA—O2B—H2BB	103 (4)
O1A—N1A—C1A—N2A	-178.9 (2)	O1B—N1B—C1B—N2B	179.6 (2)
C2A—N1A—C1A—N2A	0.2 (3)	C2B—N1B—C1B—N2B	0.0 (4)
C3A—N2A—C1A—N1A	-0.1 (3)	C3B—N2B—C1B—N1B	0.0 (3)
C4A—N2A—C1A—N1A	176.4 (3)	C4B—N2B—C1B—N1B	-174.4 (3)
C1A—N1A—C2A—C3A	-0.2 (3)	C1B—N1B—C2B—C3B	0.0 (4)
O1A—N1A—C2A—C3A	179.0 (2)	O1B—N1B—C2B—C3B	-179.6 (2)
N1A—C2A—C3A—N2A	0.1 (3)	N1B—C2B—C3B—N2B	0.0 (3)
C1A—N2A—C3A—C2A	0.0 (3)	C1B—N2B—C3B—C2B	0.0 (3)
C4A—N2A—C3A—C2A	-176.6 (3)	C4B—N2B—C3B—C2B	174.4 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2A—H2AA...O1B	1.03 (6)	1.73 (6)	2.752 (3)	172 (4)
O2A—H2AB...O1A	0.83 (5)	1.94 (5)	2.773 (3)	175 (4)
O2B—H2BA...O1B	0.83 (4)	1.94 (4)	2.752 (3)	167 (4)
O2B—H2BB...O1A ⁱ	0.94 (5)	1.86 (5)	2.790 (3)	171 (5)
C1A—H1A...O1A ⁱⁱ	0.95	2.47	3.248 (4)	139
C4A—H4AC...O1A ⁱⁱ	0.98	2.46	3.308 (4)	145
C4B—H4BC...O1A ⁱⁱ	0.98	2.56	3.336 (4)	136
C1B—H1B...O1B ⁱ	0.95	2.48	3.248 (4)	138
C2B—H2B...O2B ⁱⁱⁱ	0.95	2.41	3.298 (4)	155
C4B—H4BA...O1B ⁱ	0.98	2.50	3.345 (4)	144

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