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N^1,N^1-Dimethylpropane-1,2-diaminium bis(6-carboxypyridine-2-carboxylate) monohydrate

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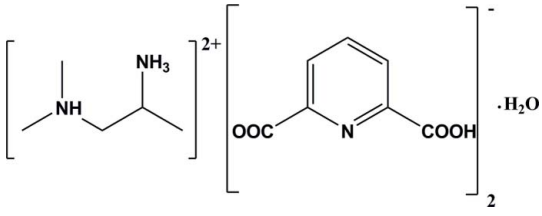
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.084; data-to-parameter ratio = 15.6.

The asymmetric unit of the title proton-transfer compound, $C_5H_{16}N_2^{2+}\cdot 2C_7H_4NO_4^-\cdot H_2O$, consists of two mono-deprotonated pyridine-2,6-dicarboxylic acid molecules as anions, *viz.* $(py\text{-}2,6\text{-}dcH)^-$, one diprotonated N^1,N^1-dimethylpropane-1,2-diamine molecule as a cation, *viz.* $(dmpdaH_2)^{2+}$, and one water molecule. The crystal packing shows extensive O—H···O, N—H···O, N—H···N and O—H···N and weak intermolecular C—H···O hydrogen bonds. These interactions link the $(dmpdaH_2)^{2+}$ cation, the $(py\text{-}2,6\text{-}dcH)^-$ anions and water molecule and play an important role in the stabilization of crystal packing.

Related literature

For background to proton-transfer compounds, see: Aghabozorg *et al.* (2008). For related structures, see: Aghabozorg, Bayan *et al.* (2011); Aghabozorg, Mofidi Rouchi *et al.* (2011); Aghabozorg, Saemi *et al.* (2011); Sharif *et al.* (2010).



Experimental

Crystal data

$C_5H_{16}N_2^{2+}\cdot 2C_7H_4NO_4^-\cdot H_2O$
M_r = 454.44
Orthorhombic, $P2_12_12_1$
a = 11.826 (8) Å
b = 13.376 (8) Å
c = 13.479 (8) Å
V = 2132 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.42 × 0.38 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.708, T_{max} = 0.746
70173 measured reflections
4650 independent reflections
4509 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.084
S = 1.07
4650 reflections
298 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O9	0.91	1.82	2.711 (2)	165
N2—H1A···O9	0.89	2.55	3.392 (3)	158
N2—H1A···N3	0.89	2.40	2.988 (2)	124
N2—H2A···O5^{i}	0.89	2.25	2.948 (2)	135
N2—H2A···N4^{i}	0.89	2.32	3.123 (3)	151
O3—H3···O5	0.82	1.68	2.473 (2)	162
N2—H3A···O2^{ii}	0.89	2.05	2.845 (2)	148
N2—H3A···O7^{i}	0.89	2.30	2.902 (2)	125
O7—H7···O1^{iii}	0.82	1.75	2.535 (2)	159
O9—H9A···O1	0.80 (2)	2.30 (2)	2.900 (2)	132 (2)
O9—H9A···N3	0.80 (2)	2.38 (2)	3.107 (3)	152 (2)
O9—H9B···O3	0.83 (2)	2.33 (3)	2.958 (3)	133 (3)
O9—H9B···O6	0.83 (2)	2.16 (2)	2.844 (2)	139 (3)
C3—H3B···O6^{iv}	0.97	2.58	3.541 (3)	172
C3—H3C···O2^{ii}	0.97	2.20	3.057 (3)	147
C4—H4···O4^{i}	0.98	2.45	3.349 (3)	152
C15—H15···O8^{v}	0.93	2.48	3.166 (3)	131
C2—H20A···O4^{i}	0.96	2.57	3.443 (3)	152

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2081).

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Acta Cryst. (2011). E**67**, o932–o933 [doi:10.1107/S1600536811009287]

N^1,N^1-Dimethylpropane-1,2-diaminium bis(6-carboxypyridine-2-carboxylate) monohydrate

Hossein Aghabozorg, Mahsa Foroughian, Alireza Foroumadi, Giuseppe Bruno and Hadi Amiri Rudbari

S1. Comment

Proton transfer is very important in chemistry, biochemistry and medicinal chemistry. In order to synthesize new types of proton transfer compounds our research group focusses on forming new compounds from pyridine dicarboxcylic acids (Aghabozorg *et al.* 2008) and different organic bases with nitrogen donor sites such as propane-1,3-diamine (Aghabozorg, Bayan *et al.*, 2011), diethylenetriamine (Aghabozorg, Saemi *et al.*, 2011) and 2-amino-4-methylpyridine (Aghabozorg, Mofidi Rouchi *et al.*, 2011; Sharif *et al.*, 2010).

The asymmetric unit of the title compound consists of two mono deprotonated pyridine-2,6-dicarboxylic acid as anion, one diprotonated *N*,*N*′-dimethyl-1,2-propanediamine as cation, and one water molecule (Fig. 1). In the crystal packing there are extensive O—H···O, N—H···O, N—H···N, O—H···N and weak intermolecular C—H···O hydrogen bonds (Table 1, Fig. 2, Fig. 3). The *N*,*N*′-dimethyl-1,2-propanediaminium $(dmpdaH_2)^{2+}$ cation is linked to the water molecule by N—H···O hydrogen bonds. This water molecule also connects the cationic part to the anionic part by N—H···O, O—H···N and O—H···O hydrogen bonds. These hydrogen bonds play an important role in the stabilization of the crystal packing.

S2. Experimental

The solution of pyridine-2,6-dicarboxylic acid (0.334 g, 2 mmol) in 15 ml water was reacted with a solution of *N*,*N*′-dimethyl-1,2-propanediamine (0.258 ml, 2 mmol) in 7 ml water in 1:1 molar ratios. The reaction mixture was stirred for 2hrs at 298 K. The colorless crystals of the title compound appeared after slow evaporation of solvent at room temperature in darkness (m.p:177°C).

S3. Refinement

The hydrogen atoms of the water molecule were found in difference Fourier map and refined isotropically with distance restraints of O—H 0.80 (2) and 0.83 (2) for H9A and H9B, respectively and a H···H distance of 1.276 (4). The other H-atoms were included at calculated positions and treated as riding atoms: O—H = 0.82 Å, N—H = 0.91, 0.89 Å for NH and NH_3, C—H = 0.93, 0.98, 0.97 and 0.96 Å for CH(aliphatic), CH(aromatic), CH_2 and CH_3 hydrogen atoms, respectively. These H-atoms were refined with *Uiso*(H) = k × *Ueq*(parent atom), where k = 1.5 for OH and CH_3 H-atoms, and k = 1.2 for all other H-atoms.

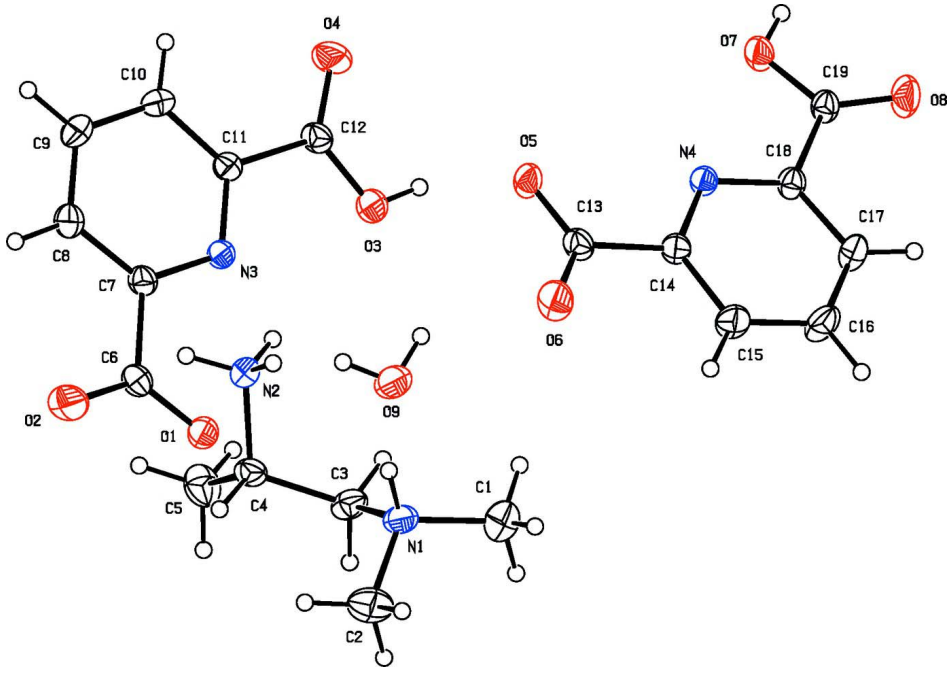

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at 30% probability level.

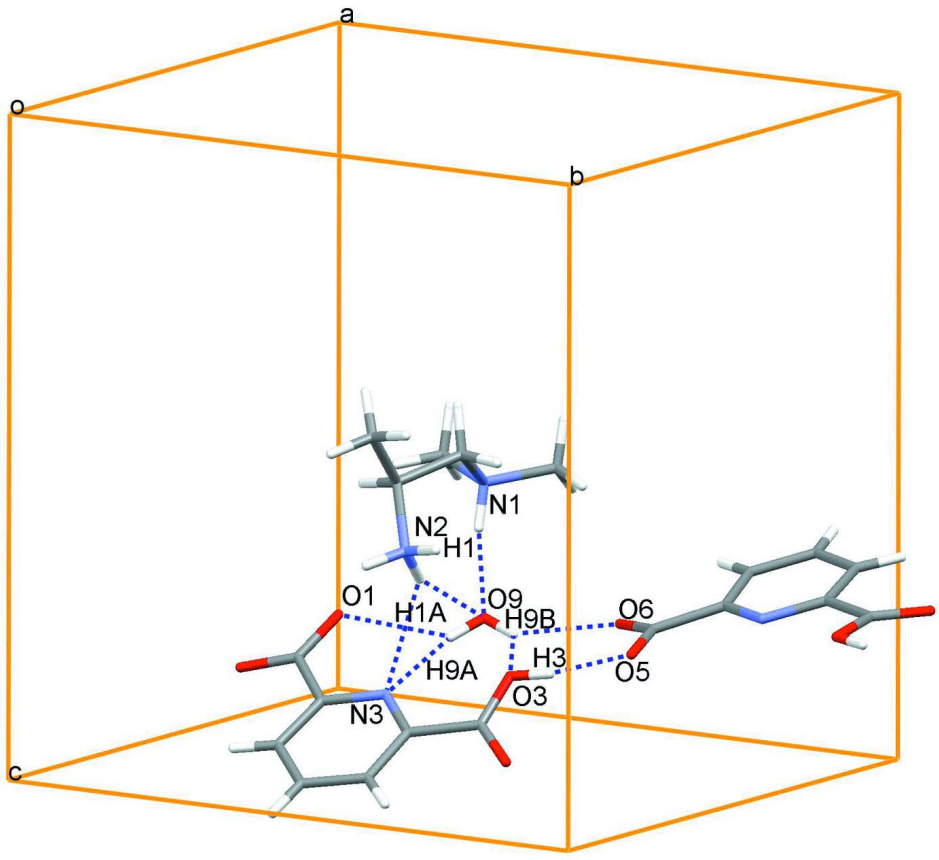

Figure 2

The packing diagram of the title compound showing hydrogen bonding interactions in the asymmetric unit as blue dashed lines.

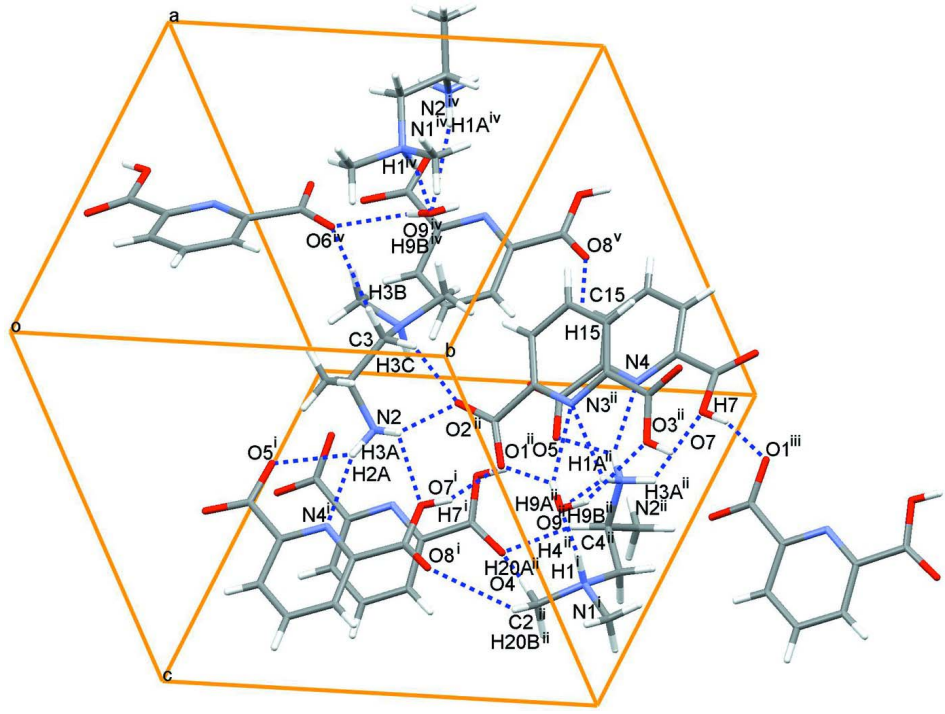

Figure 3

The packing diagram of the title compound showing all hydrogen bonding interactions as blue dashed lines. Symmetry codes: (i) -*x*+1, *y*-1/2, -*z*+3/2; (ii) -*x*+1, *y*+1/2, -*z*+3/2; (iii) *x*, *y*+1, *z*; (iv) -*x*+3/2, -*y*+1, *z*-1/2; (v) -*x*+2, *y*-1/2, -*z*+3/2.

N^1,*N*^{1}-Dimethylpropane-1,2-diaminium bis(6-carboxypyridine-2-carboxylate) monohydrate

Crystal data

$C_5H_{16}N_2^{2+}\cdot 2C_7H_4NO_4^-\cdot H_2O$
M_r = 454.44
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 11.826 (8) Å
b = 13.376 (8) Å
c = 13.479 (8) Å
V = 2132 (2) Å^3
Z = 4

F(000) = 960
D_x = 1.416 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 296(2) reflections
θ = 2.2–27.0°
μ = 0.11 mm^{-1}
T = 293 K
Irregular, colorless
0.42 × 0.38 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.708, T_{max} = 0.746

70173 measured reflections
4650 independent reflections
4509 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.0°, θ_{min} = 2.2°
h = −15→15
k = −17→17
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.084$
$S = 1.07$
4650 reflections
298 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.2616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0064 (13)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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	0.82378 (16)	0.49387 (15)	0.59189 (15)	0.0608 (4)
H10A	0.7917	0.5578	0.6078	0.091*
H10B	0.8457	0.4929	0.5233	0.091*
H10C	0.8890	0.4823	0.6328	0.091*
C2	0.78465 (16)	0.31373 (12)	0.58604 (14)	0.0589 (4)
H20A	0.7278	0.2639	0.5979	0.088*
H20B	0.8493	0.3005	0.6271	0.088*
H20C	0.8067	0.3119	0.5175	0.088*
C3	0.63379 (12)	0.43601 (10)	0.55191 (10)	0.0410 (3)
H3B	0.6481	0.4194	0.4830	0.049*
H3C	0.6194	0.5073	0.5552	0.049*
C4	0.52821 (12)	0.38176 (9)	0.58465 (9)	0.0352 (3)
H4	0.5460	0.3112	0.5963	0.042*
C5	0.44060 (16)	0.38990 (14)	0.50382 (12)	0.0557 (4)
H5A	0.3731	0.3555	0.5239	0.084*
H5B	0.4694	0.3603	0.4440	0.084*
H5C	0.4235	0.4591	0.4921	0.084*
C6	0.51400 (11)	0.21400 (9)	0.86340 (10)	0.0361 (3)
C7	0.44510 (11)	0.29707 (9)	0.91060 (9)	0.0323 (2)
C8	0.34944 (14)	0.27536 (10)	0.96579 (11)	0.0469 (3)
H8	0.3263	0.2095	0.9746	0.056*
C9	0.28930 (15)	0.35256 (12)	1.00737 (13)	0.0547 (4)
H9	0.2244	0.3398	1.0442	0.066*

C10	0.32657 (12)	0.44927 (11)	0.99364 (11)	0.0433 (3)
H10	0.2881	0.5029	1.0218	0.052*
C11	0.42230 (10)	0.46459 (9)	0.93714 (9)	0.0309 (2)
C12	0.46436 (11)	0.56979 (9)	0.91995 (9)	0.0347 (3)
C13	0.74414 (11)	0.71223 (9)	0.80029 (10)	0.0360 (3)
C14	0.81762 (10)	0.79792 (9)	0.76580 (10)	0.0327 (2)
C15	0.92660 (12)	0.77789 (11)	0.73243 (12)	0.0446 (3)
H15	0.9531	0.7125	0.7291	0.054*
C16	0.99431 (12)	0.85656 (12)	0.70449 (13)	0.0490 (4)
H16	1.0672	0.8452	0.6812	0.059*
C17	0.95259 (11)	0.95225 (11)	0.71152 (10)	0.0402 (3)
H17	0.9975	1.0069	0.6951	0.048*
C18	0.84204 (10)	0.96563 (9)	0.74366 (9)	0.0315 (2)
C19	0.79612 (10)	1.07016 (9)	0.75010 (9)	0.0330 (2)
N1	0.73847 (10)	0.41412 (9)	0.61018 (8)	0.0399 (3)
H1	0.7203	0.4150	0.6758	0.048*
N2	0.47949 (9)	0.42609 (8)	0.67685 (8)	0.0340 (2)
H1A	0.5298	0.4217	0.7258	0.051*
H2A	0.4171	0.3930	0.6936	0.051*
H3A	0.4626	0.4900	0.6661	0.051*
N3	0.48126 (8)	0.39046 (7)	0.89568 (7)	0.0291 (2)
N4	0.77430 (8)	0.89011 (7)	0.76994 (7)	0.0295 (2)
O1	0.59431 (8)	0.23905 (6)	0.80781 (8)	0.0420 (2)
O2	0.48705 (13)	0.12753 (7)	0.88430 (10)	0.0636 (4)
O3	0.54684 (9)	0.57393 (7)	0.85809 (8)	0.0454 (2)
H3	0.5668	0.6322	0.8511	0.068*
O4	0.42263 (12)	0.63994 (8)	0.96310 (10)	0.0592 (3)
O5	0.64280 (8)	0.73404 (6)	0.82184 (8)	0.0417 (2)
O6	0.78683 (10)	0.62868 (7)	0.80691 (11)	0.0570 (3)
O7	0.68767 (8)	1.07388 (7)	0.76530 (8)	0.0428 (2)
H7	0.6673	1.1324	0.7681	0.064*
O8	0.85621 (10)	1.14190 (8)	0.74214 (11)	0.0578 (3)
O9	0.72243 (10)	0.42389 (9)	0.81056 (8)	0.0489 (3)
H9A	0.6703 (19)	0.3950 (18)	0.835 (2)	0.099 (9)*
H9B	0.711 (3)	0.4830 (14)	0.827 (2)	0.115 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0545 (9)	0.0633 (10)	0.0645 (10)	−0.0123 (8)	0.0124 (8)	−0.0003 (8)
C2	0.0586 (10)	0.0474 (9)	0.0706 (10)	0.0237 (8)	0.0006 (8)	−0.0045 (8)
C3	0.0424 (7)	0.0358 (6)	0.0446 (7)	0.0094 (6)	0.0129 (5)	0.0066 (6)
C4	0.0431 (7)	0.0256 (5)	0.0370 (6)	0.0036 (5)	0.0063 (5)	−0.0008 (5)
C5	0.0664 (10)	0.0583 (9)	0.0423 (7)	−0.0145 (8)	−0.0085 (7)	−0.0005 (7)
C6	0.0419 (6)	0.0232 (5)	0.0431 (6)	0.0004 (5)	0.0016 (5)	0.0027 (5)
C7	0.0382 (6)	0.0248 (5)	0.0340 (5)	−0.0014 (5)	0.0030 (5)	0.0020 (4)
C8	0.0547 (8)	0.0329 (7)	0.0529 (8)	−0.0130 (6)	0.0179 (7)	−0.0007 (6)
C9	0.0528 (9)	0.0525 (8)	0.0587 (9)	−0.0150 (7)	0.0298 (8)	−0.0087 (7)

C10	0.0406 (7)	0.0406 (7)	0.0489 (7)	0.0015 (5)	0.0156 (6)	−0.0094 (6)
C11	0.0322 (6)	0.0276 (5)	0.0327 (5)	0.0004 (5)	0.0011 (5)	−0.0007 (4)
C12	0.0375 (6)	0.0256 (5)	0.0411 (6)	0.0034 (5)	−0.0008 (5)	−0.0004 (5)
C13	0.0369 (6)	0.0256 (5)	0.0455 (7)	0.0014 (5)	−0.0014 (5)	−0.0007 (5)
C14	0.0296 (6)	0.0273 (5)	0.0412 (6)	0.0026 (4)	0.0001 (5)	0.0010 (5)
C15	0.0341 (6)	0.0351 (6)	0.0647 (9)	0.0091 (5)	0.0054 (6)	0.0012 (6)
C16	0.0283 (6)	0.0533 (8)	0.0654 (9)	0.0069 (6)	0.0112 (6)	0.0066 (7)
C17	0.0292 (6)	0.0420 (7)	0.0495 (7)	−0.0043 (5)	0.0044 (5)	0.0083 (6)
C18	0.0294 (5)	0.0295 (6)	0.0357 (6)	−0.0009 (4)	−0.0008 (5)	0.0046 (4)
C19	0.0317 (5)	0.0283 (5)	0.0391 (6)	−0.0024 (5)	0.0002 (5)	0.0053 (5)
N1	0.0420 (6)	0.0376 (6)	0.0400 (5)	0.0045 (5)	0.0107 (5)	−0.0022 (4)
N2	0.0305 (5)	0.0362 (5)	0.0353 (5)	−0.0017 (4)	0.0039 (4)	−0.0012 (4)
N3	0.0299 (5)	0.0238 (4)	0.0335 (5)	0.0005 (4)	0.0034 (4)	0.0018 (4)
N4	0.0267 (4)	0.0255 (4)	0.0363 (5)	0.0006 (4)	0.0008 (4)	0.0017 (4)
O1	0.0368 (4)	0.0260 (4)	0.0632 (6)	0.0017 (4)	0.0110 (4)	−0.0009 (4)
O2	0.0880 (9)	0.0216 (4)	0.0814 (8)	0.0001 (5)	0.0362 (7)	0.0061 (5)
O3	0.0495 (5)	0.0229 (4)	0.0637 (6)	−0.0027 (4)	0.0157 (5)	0.0024 (4)
O4	0.0746 (8)	0.0288 (5)	0.0742 (8)	0.0032 (5)	0.0236 (7)	−0.0083 (5)
O5	0.0334 (4)	0.0256 (4)	0.0660 (6)	−0.0011 (4)	0.0090 (4)	0.0041 (4)
O6	0.0486 (6)	0.0267 (5)	0.0956 (9)	0.0067 (4)	0.0086 (6)	0.0069 (5)
O7	0.0325 (4)	0.0244 (4)	0.0714 (6)	0.0015 (3)	0.0023 (4)	−0.0022 (4)
O8	0.0448 (6)	0.0313 (5)	0.0973 (9)	−0.0083 (4)	0.0141 (6)	0.0070 (5)
O9	0.0454 (6)	0.0497 (6)	0.0516 (6)	0.0039 (5)	0.0127 (5)	−0.0008 (5)

Geometric parameters (Å, º)

C1—N1	1.489 (2)	C10—H10	0.9300
C1—H10A	0.9600	C11—N3	1.3347 (15)
C1—H10B	0.9600	C11—C12	1.5104 (16)
C1—H10C	0.9600	C12—O4	1.2094 (16)
C2—N1	1.4857 (18)	C12—O3	1.2843 (16)
C2—H20A	0.9600	C13—O6	1.2296 (16)
C2—H20B	0.9600	C13—O5	1.2672 (16)
C2—H20C	0.9600	C13—C14	1.5117 (17)
C3—N1	1.4950 (19)	C14—N4	1.3364 (15)
C3—C4	1.5101 (18)	C14—C15	1.3910 (18)
C3—H3B	0.9700	C15—C16	1.375 (2)
C3—H3C	0.9700	C15—H15	0.9300
C4—N2	1.4926 (15)	C16—C17	1.375 (2)
C4—C5	1.507 (2)	C16—H16	0.9300
C4—H4	0.9800	C17—C18	1.3889 (17)
C5—H5A	0.9600	C17—H17	0.9300
C5—H5B	0.9600	C18—N4	1.3371 (15)
C5—H5C	0.9600	C18—C19	1.5025 (16)
C6—O2	1.2323 (16)	C19—O8	1.1988 (16)
C6—O1	1.2553 (16)	C19—O7	1.2996 (15)
C6—C7	1.5177 (17)	N1—H1	0.9100
C7—N3	1.3356 (15)	N2—H1A	0.8900

C7—C8	1.3848 (18)	N2—H2A	0.8900
C8—C9	1.373 (2)	N2—H3A	0.8900
C8—H8	0.9300	O3—H3	0.8200
C9—C10	1.379 (2)	O7—H7	0.8200
C9—H9	0.9300	O9—H9A	0.799 (17)
C10—C11	1.3798 (18)	O9—H9B	0.830 (17)

N1—C1—H10A	109.5	N3—C11—C10	123.33 (11)
N1—C1—H10B	109.5	N3—C11—C12	117.12 (10)
H10A—C1—H10B	109.5	C10—C11—C12	119.55 (11)
N1—C1—H10C	109.5	O4—C12—O3	126.07 (13)
H10A—C1—H10C	109.5	O4—C12—C11	120.98 (12)
H10B—C1—H10C	109.5	O3—C12—C11	112.94 (10)
N1—C2—H20A	109.5	O6—C13—O5	125.52 (13)
N1—C2—H20B	109.5	O6—C13—C14	118.39 (12)
H20A—C2—H20B	109.5	O5—C13—C14	116.08 (11)
N1—C2—H20C	109.5	N4—C14—C15	123.12 (12)
H20A—C2—H20C	109.5	N4—C14—C13	117.80 (11)
H20B—C2—H20C	109.5	C15—C14—C13	119.08 (11)
N1—C3—C4	115.91 (11)	C16—C15—C14	118.73 (13)
N1—C3—H3B	108.3	C16—C15—H15	120.6
C4—C3—H3B	108.3	C14—C15—H15	120.6
N1—C3—H3C	108.3	C15—C16—C17	118.99 (13)
C4—C3—H3C	108.3	C15—C16—H16	120.5
H3B—C3—H3C	107.4	C17—C16—H16	120.5
N2—C4—C5	107.93 (12)	C16—C17—C18	118.63 (12)
N2—C4—C3	111.82 (11)	C16—C17—H17	120.7
C5—C4—C3	108.80 (11)	C18—C17—H17	120.7
N2—C4—H4	109.4	N4—C18—C17	123.32 (12)
C5—C4—H4	109.4	N4—C18—C19	118.11 (10)
C3—C4—H4	109.4	C17—C18—C19	118.57 (11)
C4—C5—H5A	109.5	O8—C19—O7	124.64 (12)
C4—C5—H5B	109.5	O8—C19—C18	121.69 (11)
H5A—C5—H5B	109.5	O7—C19—C18	113.67 (10)
C4—C5—H5C	109.5	C2—N1—C1	111.23 (13)
H5A—C5—H5C	109.5	C2—N1—C3	111.49 (12)
H5B—C5—H5C	109.5	C1—N1—C3	109.50 (12)
O2—C6—O1	125.64 (13)	C2—N1—H1	108.2
O2—C6—C7	116.91 (12)	C1—N1—H1	108.2
O1—C6—C7	117.45 (10)	C3—N1—H1	108.2
N3—C7—C8	122.59 (12)	C4—N2—H1A	109.5
N3—C7—C6	116.73 (11)	C4—N2—H2A	109.5
C8—C7—C6	120.68 (11)	H1A—N2—H2A	109.5
C9—C8—C7	118.99 (12)	C4—N2—H3A	109.5
C9—C8—H8	120.5	H1A—N2—H3A	109.5
C7—C8—H8	120.5	H2A—N2—H3A	109.5
C8—C9—C10	119.01 (13)	C11—N3—C7	117.68 (10)
C8—C9—H9	120.5	C14—N4—C18	117.15 (10)

C10—C9—H9	120.5	C12—O3—H3	109.5
C9—C10—C11	118.40 (12)	C19—O7—H7	109.5
C9—C10—H10	120.8	H9A—O9—H9B	103 (3)
C11—C10—H10	120.8		
N1—C3—C4—N2	75.97 (14)	N4—C14—C15—C16	1.6 (2)
N1—C3—C4—C5	−164.91 (12)	C13—C14—C15—C16	−177.73 (14)
O2—C6—C7—N3	173.61 (14)	C14—C15—C16—C17	0.7 (2)
O1—C6—C7—N3	−5.34 (18)	C15—C16—C17—C18	−2.0 (2)
O2—C6—C7—C8	−6.7 (2)	C16—C17—C18—N4	1.2 (2)
O1—C6—C7—C8	174.39 (14)	C16—C17—C18—C19	−179.26 (13)
N3—C7—C8—C9	−0.3 (2)	N4—C18—C19—O8	168.74 (14)
C6—C7—C8—C9	179.99 (14)	C17—C18—C19—O8	−10.81 (19)
C7—C8—C9—C10	−0.6 (3)	N4—C18—C19—O7	−10.92 (16)
C8—C9—C10—C11	1.0 (3)	C17—C18—C19—O7	169.53 (12)
C9—C10—C11—N3	−0.5 (2)	C4—C3—N1—C2	74.31 (15)
C9—C10—C11—C12	179.35 (14)	C4—C3—N1—C1	−162.17 (12)
N3—C11—C12—O4	−172.38 (13)	C10—C11—N3—C7	−0.30 (18)
C10—C11—C12—O4	7.7 (2)	C12—C11—N3—C7	179.80 (11)
N3—C11—C12—O3	6.62 (16)	C8—C7—N3—C11	0.73 (19)
C10—C11—C12—O3	−173.29 (12)	C6—C7—N3—C11	−179.55 (11)
O6—C13—C14—N4	−172.34 (13)	C15—C14—N4—C18	−2.43 (19)
O5—C13—C14—N4	6.38 (18)	C13—C14—N4—C18	176.91 (11)
O6—C13—C14—C15	7.0 (2)	C17—C18—N4—C14	1.00 (18)
O5—C13—C14—C15	−174.25 (13)	C19—C18—N4—C14	−178.52 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O9	0.91	1.82	2.711 (2)	165
N2—H1*A*···O9	0.89	2.55	3.392 (3)	158
N2—H1*A*···N3	0.89	2.40	2.988 (2)	124
N2—H2*A*···O5^{i}	0.89	2.25	2.948 (2)	135
N2—H2*A*···N4^{i}	0.89	2.32	3.123 (3)	151
O3—H3···O5	0.82	1.68	2.473 (2)	162
N2—H3*A*···O2^{ii}	0.89	2.05	2.845 (2)	148
N2—H3*A*···O7^{i}	0.89	2.30	2.902 (2)	125
O7—H7···O1^{iii}	0.82	1.75	2.535 (2)	159
O9—H9*A*···O1	0.80 (2)	2.30 (2)	2.900 (2)	132 (2)
O9—H9*A*···N3	0.80 (2)	2.38 (2)	3.107 (3)	152 (2)
O9—H9*B*···O3	0.83 (2)	2.33 (3)	2.958 (3)	133 (3)
O9—H9*B*···O6	0.83 (2)	2.16 (2)	2.844 (2)	139 (3)
C3—H3*B*···O6^{iv}	0.97	2.58	3.541 (3)	172
C3—H3*C*···O2^{ii}	0.97	2.20	3.057 (3)	147
C4—H4···O4^{i}	0.98	2.45	3.349 (3)	152

C15—H15···O8^{v}	0.93	2.48	3.166 (3)	131
C2—H20*A*···O4^{i}	0.96	2.57	3.443 (3)	152

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) *x*, *y*+1, *z*; (iv) −*x*+3/2, −*y*+1, *z*−1/2; (v) −*x*+2, *y*−1/2, −*z*+3/2.