

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis[8-ethyl-5-oxo-2-(piperazin-4-ium-1-yl)-5,8-dihydropyrido[2,3-*d*]pyrimidine-6-carboxylic acid] 2,5-dicarboxybenzene-1,4-dicarboxylate octahydrate

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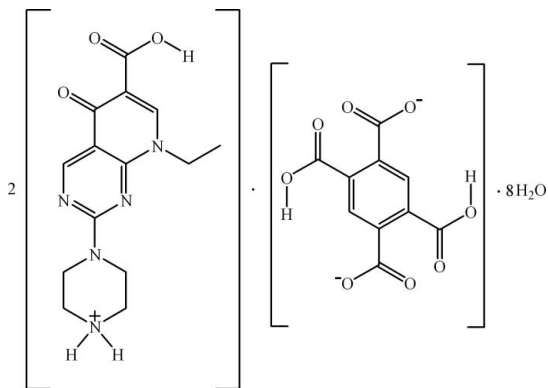
Received 19 March 2011; accepted 25 March 2011

 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.046; wR factor = 0.160; data-to-parameter ratio = 14.4.

The asymmetric unit of the title compound, $2\text{C}_{14}\text{H}_{18}\text{N}_5\text{O}_3^+ \cdot \text{C}_{10}\text{H}_5\text{O}_8^{2-} \cdot 8\text{H}_2\text{O}$, contains one $[\text{H}_2\text{ppa}]^+$ cation, one half of an $[\text{H}_2\text{btcc}]^{2-}$ anion ($\text{H}_4\text{btcc} = 1,2,4,5$ -benzenetetracarboxylic acid and $\text{Hppa} = 8$ -ethyl-5-oxo-2-piperazin-1-yl-5,8-dihydropyrido[2,3-*d*]pyrimidine-6-carboxylic acid) that is completed by inversion symmetry and four water molecules. In the crystal, the molecules are connected by intermolecular hydrogen-bonding interactions and π - π stacking between the benzene rings of the $[\text{H}_2\text{btcc}]^{2-}$ anion and the pyrimidine rings of the $[\text{H}_2\text{ppa}]^+$ cation [centroid-centroid distance = 3.597 (3) Å], generating a three-dimensional supramolecular structure.

Related literature

For general background to the use of quinolones in the treatment of infections, see: Mizuki *et al.* (1996).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{18}\text{N}_5\text{O}_3^+ \cdot 0.5\text{C}_{10}\text{H}_5\text{O}_8^{2-} \cdot 4\text{H}_2\text{O}$
 $M_r = 502.46$
 Triclinic, $P\bar{1}$
 $a = 8.8336$ (16) Å
 $b = 11.103$ (2) Å
 $c = 12.445$ (2) Å
 $\alpha = 83.010$ (2)°
 $\beta = 76.737$ (2)°

$\gamma = 73.831$ (2)°
 $V = 1138.9$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹
 $T = 296$ K
 $0.52 \times 0.48 \times 0.39$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.940$, $T_{\max} = 0.954$

10252 measured reflections
 5071 independent reflections
 3550 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.160$
 $S = 1.01$
 5071 reflections
 352 parameters
 14 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O6—H6A···O4 ⁱ	0.92 (2)	1.47 (2)	2.392 (2)	178 (3)
N1—H1A···OW1 ⁱⁱ	0.90	2.08	2.952 (2)	164
N1—H1A···O6 ⁱⁱ	0.90	2.56	3.022 (2)	113
N1—H1B···OW2 ⁱⁱ	0.90	1.82	2.717 (2)	176
OW1—HW1A···O1 ⁱⁱⁱ	0.88 (2)	1.98 (2)	2.780 (2)	150 (2)
OW1—HW1B···O3	0.85 (2)	2.38 (2)	3.041 (2)	135 (2)
OW1—HW1B···O7	0.85 (2)	2.57 (2)	3.204 (2)	132 (2)
OW1—HW1B···O6	0.85 (2)	2.59 (2)	3.084 (2)	118 (2)
O2—H2A···O3	0.99 (2)	1.56 (2)	2.5013 (19)	159 (2)
OW2—HW2B···OW4 ^{iv}	0.86 (2)	1.86 (2)	2.703 (3)	168 (3)
OW2—HW2A···O5 ^{iv}	0.84 (2)	1.98 (2)	2.819 (2)	173 (3)
OW3—HW3A···OW1 ^v	0.85 (2)	1.93 (2)	2.770 (2)	169 (3)
OW3—HW3B···O2 ^{vi}	0.85 (2)	2.17 (2)	3.011 (3)	172 (3)
OW4—HW4A···O4 ^{vii}	0.87 (2)	1.94 (2)	2.782 (2)	163 (3)
OW4—HW4B···OW3	0.81 (2)	1.97 (2)	2.775 (3)	174 (3)

Symmetry codes: (i) $-x, -y + 2, -z + 1$; (ii) $x, y, z - 1$; (iii) $x + 1, y, z$; (iv) $-x + 1, -y + 2, -z + 1$; (v) $-x + 1, -y + 1, -z + 1$; (vi) $x + 1, y, z - 1$; (vii) $-x + 1, -y + 2, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Science and Technology Foundation of Southwest University (SWUB2007035).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2005).

References

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supporting information

Acta Cryst. (2011). E67, o1011–o1012 [doi:10.1107/S1600536811011068]

Bis[8-ethyl-5-oxo-2-(piperazin-4-ium-1-yl)-5,8-dihydropyrido[2,3-d]pyrimidine-6-carboxylic acid] 2,5-dicarboxybenzene-1,4-dicarboxylate octahydrate

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S1. Comment

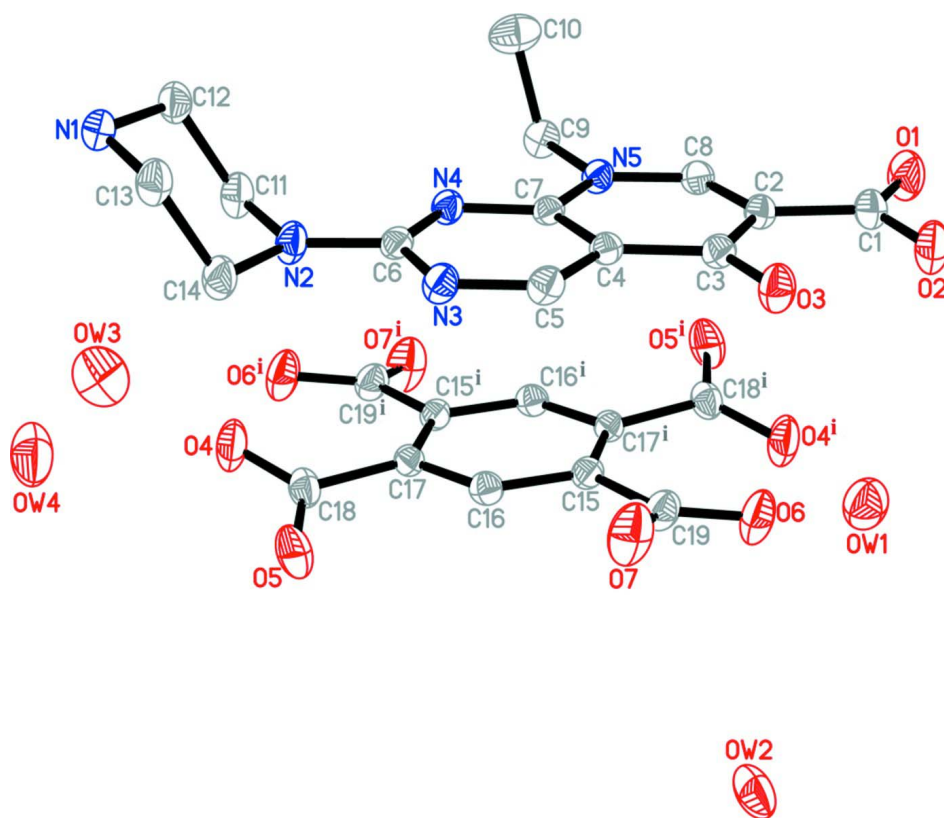
Pipemidic acid (8-ethyl-5-oxo-2-piperazin-1-yl-5,8-dihydropyrido[2,3-d]pyrimidine-6-carboxylic acid) is member of quinolones used to treat infections (Mizuki *et al.*, 1996). The complexes of the Hppa and H₄btec have not been reported till now. In this paper, the structure of the title compound, **1**, is described (Fig. 1). The asymmetric unit contains one [H₂ppa]⁺cation, one half of [H₂btec]²⁻ anion that is completed by inversion symmetry, and four lattice water molecules. The molecules are linked by intermolecular N—H···O and O—H···O hydrogen-bonding interactions (N···O = 2.717 (2)–3.022 (2) Å, O···O = 2.392 (2)–3.204 (2) Å) and π — π stacking between the benzene rings of [H₂btec]²⁻ anion and the pyrimidine rings of [H₂ppa]⁺cation (centroid distance of 3.597 (3) Å) to form a three-dimensional supramolecular structure.

S2. Experimental

A mixture of AgNO₃ (0.085 g, 0.5 mmol), Hppa (0.089 g, 0.25 mmol), H₄btec (0.064 g, 0.25 mmol) and distilled water (8 ml) was stirred for 20 min. in air. The mixture was then transferred to a 18 ml Teflon-lined hydrothermal bomb. The bomb was kept at 393 K for 96 h under autogenous pressure. Upon cooling, colorless blocks of **1** were obtained from the reaction mixture.

S3. Refinement

The H atoms bonded to C atoms were positioned geometrically and refined using a riding model approximation [aromatic C—H = 0.93 Å, aliphatic C—H = 0.96–0.97 Å], with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$. The H on N atoms were located in difference Fourier maps, and refined with distances restraint of N—H = 0.90 Å, and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{N})$. The H atoms bonded to O atoms were located in difference Fourier maps and refined with $U_{\text{iso}}(\text{H}) = 1.3 U_{\text{eq}}(\text{O})$ for carboxyl groups of [C₁₄H₁₈O₃N₅]⁺ and [C₁₀H₄O₈]²⁻ respectively. The O—H bonds are 0.986 Å and 0.924 Å in carboxyl groups of [C₁₄H₁₈O₃N₅]⁺ and [C₁₀H₄O₈]²⁻. The H atoms bonded to OW atoms were located in a difference Fourier maps and refined with OW—H = 0.812 Å–0.878 Å and $U_{\text{iso}}(\text{H}) = 1.1\text{--}1.5 U_{\text{eq}}(\text{OW})$.

**Figure 1**

The structure of **1**. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: $-x, -y+2, -z+1$].

Bis[8-ethyl-5-oxo-2-(piperazin-4-ium-1-yl)-5,8-dihydropyrido[2,3-*d*]pyrimidine-6-carboxylic acid] 2,5-dicarboxybenzene-1,4-dicarboxylate octahydrate

Crystal data

$C_{14}H_{18}N_5O_3^+ \cdot 0.5C_{10}H_4O_8^- \cdot 4H_2O$

$M_r = 502.46$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.8336$ (16) Å

$b = 11.103$ (2) Å

$c = 12.445$ (2) Å

$\alpha = 83.010$ (2)°

$\beta = 76.737$ (2)°

$\gamma = 73.831$ (2)°

$V = 1138.9$ (4) Å³

$Z = 2$

$F(000) = 530$

$D_x = 1.465$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 10252 reflections

$\theta = 2.5$ – 27.4 °

$\mu = 0.12$ mm⁻¹

$T = 296$ K

Block, colourless

$0.52 \times 0.48 \times 0.39$ mm

Data collection

Bruker APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.940$, $T_{\max} = 0.954$

10252 measured reflections

5071 independent reflections

3550 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 27.4^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -11 \rightarrow 11$

$k = -14 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.160$
 $S = 1.01$
 5071 reflections
 352 parameters
 14 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
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.2298 (2)	0.58949 (18)	0.76095 (14)	0.0453 (4)
C2	-0.1629 (2)	0.63420 (16)	0.64826 (13)	0.0362 (4)
C3	0.0071 (2)	0.60173 (15)	0.60569 (13)	0.0348 (4)
C4	0.05712 (18)	0.65003 (14)	0.49440 (12)	0.0316 (3)
C5	0.2186 (2)	0.62709 (16)	0.43913 (13)	0.0377 (4)
H5A	0.2968	0.5782	0.4765	0.045*
C6	0.14597 (19)	0.74223 (16)	0.28730 (13)	0.0336 (4)
C7	-0.05440 (18)	0.72426 (14)	0.43356 (12)	0.0295 (3)
C8	-0.2657 (2)	0.70738 (16)	0.58476 (13)	0.0360 (4)
H8A	-0.3755	0.7269	0.6149	0.043*
C9	-0.33740 (19)	0.83037 (17)	0.41760 (14)	0.0402 (4)
H9A	-0.2943	0.8957	0.3727	0.048*
H9B	-0.4340	0.8707	0.4687	0.048*
C10	-0.3799 (3)	0.7527 (2)	0.3443 (2)	0.0640 (6)
H10A	-0.4572	0.8056	0.3042	0.096*
H10B	-0.4251	0.6892	0.3887	0.096*
H10C	-0.2847	0.7134	0.2931	0.096*
C11	0.0817 (2)	0.85693 (19)	0.11400 (14)	0.0470 (4)
H11A	0.1022	0.9380	0.0881	0.056*
H11B	-0.0279	0.8712	0.1564	0.056*
C12	0.1010 (2)	0.78182 (19)	0.01646 (14)	0.0472 (5)

H12A	0.0685	0.7049	0.0421	0.057*
H12B	0.0317	0.8298	-0.0325	0.057*
C13	0.3844 (2)	0.68542 (19)	0.02854 (14)	0.0475 (5)
H13A	0.4945	0.6720	-0.0128	0.057*
H13B	0.3654	0.6040	0.0552	0.057*
C14	0.3613 (2)	0.7624 (2)	0.12515 (15)	0.0474 (5)
H14A	0.4303	0.7166	0.1751	0.057*
H14B	0.3910	0.8403	0.0992	0.057*
C15	0.0728 (2)	0.91872 (15)	0.58174 (12)	0.0335 (4)
C16	0.1614 (2)	0.95494 (15)	0.48134 (14)	0.0350 (4)
H16	0.271 (2)	0.9248 (16)	0.4737 (15)	0.037 (5)*
C17	0.09561 (19)	1.03470 (15)	0.39901 (12)	0.0327 (4)
C18	0.2204 (2)	1.06072 (16)	0.29829 (14)	0.0419 (4)
C19	0.1758 (2)	0.82880 (18)	0.65566 (15)	0.0459 (4)
N1	0.27157 (18)	0.75043 (15)	-0.04528 (11)	0.0452 (4)
H1A	0.2821	0.7007	-0.0999	0.054*
H1B	0.2974	0.8215	-0.0764	0.054*
N2	0.19315 (17)	0.79002 (15)	0.18387 (11)	0.0430 (4)
N3	0.26607 (16)	0.67052 (14)	0.33790 (11)	0.0396 (3)
N4	-0.01218 (15)	0.77067 (13)	0.33069 (10)	0.0338 (3)
N5	-0.21691 (15)	0.75273 (13)	0.48113 (11)	0.0333 (3)
O1	-0.37365 (18)	0.61061 (16)	0.80048 (11)	0.0640 (4)
OW1	0.34139 (19)	0.54945 (15)	0.80171 (12)	0.0614 (4)
HW1A	0.4401 (19)	0.557 (2)	0.7779 (18)	0.072*
HW1B	0.298 (2)	0.581 (2)	0.7463 (16)	0.070*
O2	-0.12330 (19)	0.52214 (15)	0.81832 (12)	0.0622 (4)
H2A	-0.018 (2)	0.516 (2)	0.7683 (19)	0.083 (8)*
OW2	0.3559 (2)	0.95983 (17)	0.85270 (14)	0.0721 (5)
HW2A	0.442 (3)	0.954 (3)	0.8054 (18)	0.097*
HW2B	0.353 (3)	1.013 (2)	0.8988 (19)	0.103*
O3	0.10748 (15)	0.53510 (12)	0.66146 (10)	0.0502 (3)
OW3	0.8262 (3)	0.59914 (18)	0.05037 (17)	0.0835 (5)
HW3A	0.764 (3)	0.562 (3)	0.0954 (17)	0.092*
HW3B	0.840 (4)	0.570 (3)	-0.0121 (15)	0.118*
OW4	0.6900 (2)	0.85376 (17)	0.01290 (14)	0.0782 (5)
HW4A	0.713 (4)	0.876 (2)	-0.0572 (15)	0.107*
HW4B	0.725 (3)	0.7783 (16)	0.022 (2)	0.092*
O4	0.17347 (18)	1.10598 (16)	0.20881 (11)	0.0655 (4)
O5	0.36177 (16)	1.03613 (14)	0.30721 (12)	0.0604 (4)
O6	0.1120 (2)	0.80744 (16)	0.75738 (11)	0.0660 (4)
H6A	0.002 (2)	0.842 (2)	0.772 (2)	0.084 (8)*
O7	0.31545 (18)	0.77853 (16)	0.61765 (13)	0.0728 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0499 (11)	0.0524 (11)	0.0330 (9)	-0.0199 (9)	-0.0005 (8)	-0.0002 (8)
C2	0.0399 (9)	0.0389 (9)	0.0298 (8)	-0.0142 (7)	-0.0017 (7)	-0.0034 (7)

C3	0.0377 (9)	0.0347 (8)	0.0327 (8)	-0.0095 (7)	-0.0074 (7)	-0.0035 (6)
C4	0.0312 (8)	0.0334 (8)	0.0311 (8)	-0.0097 (6)	-0.0052 (6)	-0.0039 (6)
C5	0.0305 (8)	0.0460 (10)	0.0352 (9)	-0.0059 (7)	-0.0080 (7)	-0.0036 (7)
C6	0.0310 (8)	0.0433 (9)	0.0284 (8)	-0.0123 (7)	-0.0040 (6)	-0.0072 (7)
C7	0.0283 (8)	0.0337 (8)	0.0280 (7)	-0.0097 (6)	-0.0042 (6)	-0.0068 (6)
C8	0.0308 (8)	0.0441 (10)	0.0324 (8)	-0.0124 (7)	0.0007 (6)	-0.0076 (7)
C9	0.0274 (8)	0.0474 (10)	0.0416 (9)	-0.0031 (7)	-0.0069 (7)	-0.0030 (7)
C10	0.0566 (13)	0.0637 (13)	0.0801 (15)	-0.0067 (10)	-0.0388 (12)	-0.0093 (11)
C11	0.0415 (10)	0.0579 (11)	0.0347 (9)	-0.0089 (8)	-0.0026 (7)	0.0040 (8)
C12	0.0452 (10)	0.0649 (12)	0.0352 (9)	-0.0233 (9)	-0.0109 (8)	0.0091 (8)
C13	0.0413 (10)	0.0584 (12)	0.0375 (10)	-0.0115 (9)	-0.0004 (8)	-0.0009 (8)
C14	0.0329 (9)	0.0750 (13)	0.0337 (9)	-0.0191 (9)	0.0004 (7)	-0.0023 (8)
C15	0.0365 (9)	0.0342 (8)	0.0304 (8)	-0.0091 (7)	-0.0076 (6)	-0.0026 (6)
C16	0.0298 (8)	0.0377 (9)	0.0365 (9)	-0.0083 (7)	-0.0045 (7)	-0.0047 (7)
C17	0.0368 (9)	0.0324 (8)	0.0288 (8)	-0.0115 (7)	-0.0022 (6)	-0.0045 (6)
C18	0.0458 (10)	0.0395 (9)	0.0358 (9)	-0.0133 (8)	0.0042 (7)	-0.0034 (7)
C19	0.0483 (11)	0.0515 (11)	0.0385 (10)	-0.0105 (9)	-0.0161 (8)	0.0025 (8)
N1	0.0549 (9)	0.0520 (9)	0.0302 (7)	-0.0205 (7)	-0.0041 (6)	-0.0018 (6)
N2	0.0332 (8)	0.0654 (10)	0.0276 (7)	-0.0139 (7)	-0.0012 (6)	0.0003 (7)
N3	0.0296 (7)	0.0550 (9)	0.0329 (7)	-0.0097 (6)	-0.0051 (6)	-0.0026 (6)
N4	0.0301 (7)	0.0423 (8)	0.0284 (7)	-0.0104 (6)	-0.0035 (5)	-0.0029 (6)
N5	0.0282 (7)	0.0410 (8)	0.0296 (7)	-0.0082 (6)	-0.0036 (5)	-0.0040 (5)
O1	0.0535 (9)	0.0927 (11)	0.0404 (7)	-0.0267 (8)	0.0077 (6)	0.0014 (7)
OW1	0.0568 (9)	0.0730 (10)	0.0537 (9)	-0.0144 (8)	-0.0185 (7)	0.0068 (7)
O2	0.0631 (10)	0.0786 (10)	0.0395 (7)	-0.0200 (8)	-0.0073 (7)	0.0145 (7)
OW2	0.0719 (11)	0.0748 (11)	0.0624 (10)	-0.0340 (9)	0.0177 (8)	-0.0015 (8)
O3	0.0469 (8)	0.0571 (8)	0.0416 (7)	-0.0086 (6)	-0.0123 (6)	0.0107 (6)
OW3	0.0943 (14)	0.0840 (13)	0.0773 (12)	-0.0392 (11)	-0.0119 (11)	0.0024 (10)
OW4	0.0926 (13)	0.0772 (11)	0.0517 (9)	-0.0234 (10)	0.0125 (9)	-0.0031 (9)
O4	0.0650 (10)	0.0936 (12)	0.0312 (7)	-0.0228 (8)	-0.0001 (6)	0.0087 (7)
O5	0.0412 (8)	0.0692 (9)	0.0569 (8)	-0.0126 (7)	0.0083 (6)	0.0090 (7)
O6	0.0667 (10)	0.0878 (11)	0.0363 (7)	-0.0113 (9)	-0.0163 (7)	0.0145 (7)
O7	0.0492 (9)	0.0929 (12)	0.0579 (9)	0.0087 (8)	-0.0165 (7)	0.0124 (8)

Geometric parameters (Å, °)

C1—O1	1.219 (2)	C12—H12B	0.9700
C1—O2	1.320 (2)	C13—N1	1.489 (2)
C1—C2	1.475 (2)	C13—C14	1.503 (3)
C2—C8	1.368 (2)	C13—H13A	0.9700
C2—C3	1.430 (2)	C13—H13B	0.9700
C3—O3	1.268 (2)	C14—N2	1.462 (2)
C3—C4	1.439 (2)	C14—H14A	0.9700
C4—C7	1.400 (2)	C14—H14B	0.9700
C4—C5	1.401 (2)	C15—C16	1.393 (2)
C5—N3	1.311 (2)	C15—C17 ⁱ	1.406 (2)
C5—H5A	0.9300	C15—C19	1.525 (2)
C6—N4	1.339 (2)	C16—C17	1.392 (2)

C6—N2	1.350 (2)	C16—H16	0.917 (19)
C6—N3	1.367 (2)	C17—C15 ⁱ	1.406 (2)
C7—N4	1.3307 (19)	C17—C18	1.525 (2)
C7—N5	1.3834 (19)	C18—O5	1.230 (2)
C8—N5	1.345 (2)	C18—O4	1.275 (2)
C8—H8A	0.9300	C19—O7	1.213 (2)
C9—N5	1.487 (2)	C19—O6	1.285 (2)
C9—C10	1.499 (3)	N1—H1A	0.9000
C9—H9A	0.9700	N1—H1B	0.9000
C9—H9B	0.9700	OW1—HW1A	0.878 (15)
C10—H10A	0.9600	OW1—HW1B	0.854 (15)
C10—H10B	0.9600	O2—H2A	0.986 (16)
C10—H10C	0.9600	OW2—HW2A	0.842 (16)
C11—N2	1.451 (2)	OW2—HW2B	0.860 (16)
C11—C12	1.507 (3)	OW3—HW3A	0.849 (16)
C11—H11A	0.9700	OW3—HW3B	0.850 (17)
C11—H11B	0.9700	OW4—HW4A	0.871 (16)
C12—N1	1.491 (2)	OW4—HW4B	0.812 (16)
C12—H12A	0.9700	O6—H6A	0.924 (17)
O1—C1—O2	120.65 (17)	N1—C13—C14	110.52 (15)
O1—C1—C2	123.66 (18)	N1—C13—H13A	109.5
O2—C1—C2	115.69 (16)	C14—C13—H13A	109.5
C8—C2—C3	120.28 (14)	N1—C13—H13B	109.5
C8—C2—C1	119.08 (16)	C14—C13—H13B	109.5
C3—C2—C1	120.64 (15)	H13A—C13—H13B	108.1
O3—C3—C2	122.75 (15)	N2—C14—C13	109.98 (14)
O3—C3—C4	121.96 (15)	N2—C14—H14A	109.7
C2—C3—C4	115.29 (14)	C13—C14—H14A	109.7
C7—C4—C5	115.19 (14)	N2—C14—H14B	109.7
C7—C4—C3	121.64 (14)	C13—C14—H14B	109.7
C5—C4—C3	123.17 (15)	H14A—C14—H14B	108.2
N3—C5—C4	123.88 (15)	C16—C15—C17 ⁱ	117.61 (15)
N3—C5—H5A	118.1	C16—C15—C19	113.70 (15)
C4—C5—H5A	118.1	C17 ⁱ —C15—C19	128.68 (15)
N4—C6—N2	117.33 (15)	C17—C16—C15	124.81 (15)
N4—C6—N3	126.49 (14)	C17—C16—H16	120.7 (11)
N2—C6—N3	116.17 (14)	C15—C16—H16	114.4 (11)
N4—C7—N5	117.27 (13)	C16—C17—C15 ⁱ	117.58 (14)
N4—C7—C4	123.16 (14)	C16—C17—C18	113.93 (15)
N5—C7—C4	119.57 (14)	C15 ⁱ —C17—C18	128.49 (15)
N5—C8—C2	123.73 (15)	O5—C18—O4	123.21 (16)
N5—C8—H8A	118.1	O5—C18—C17	118.33 (16)
C2—C8—H8A	118.1	O4—C18—C17	118.46 (16)
N5—C9—C10	111.63 (15)	O7—C19—O6	121.26 (18)
N5—C9—H9A	109.3	O7—C19—C15	119.71 (17)
C10—C9—H9A	109.3	O6—C19—C15	119.03 (17)
N5—C9—H9B	109.3	C13—N1—C12	111.63 (13)

C10—C9—H9B	109.3	C13—N1—H1A	109.3
H9A—C9—H9B	108.0	C12—N1—H1A	109.3
C9—C10—H10A	109.5	C13—N1—H1B	109.3
C9—C10—H10B	109.5	C12—N1—H1B	109.3
H10A—C10—H10B	109.5	H1A—N1—H1B	108.0
C9—C10—H10C	109.5	C6—N2—C11	123.41 (14)
H10A—C10—H10C	109.5	C6—N2—C14	122.94 (15)
H10B—C10—H10C	109.5	C11—N2—C14	112.99 (13)
N2—C11—C12	110.08 (15)	C5—N3—C6	115.45 (14)
N2—C11—H11A	109.6	C7—N4—C6	115.83 (14)
C12—C11—H11A	109.6	C8—N5—C7	119.48 (14)
N2—C11—H11B	109.6	C8—N5—C9	120.08 (13)
C12—C11—H11B	109.6	C7—N5—C9	120.43 (13)
H11A—C11—H11B	108.2	HW1A—OW1—HW1B	101.7 (17)
N1—C12—C11	110.68 (14)	C1—O2—H2A	105.2 (15)
N1—C12—H12A	109.5	HW2A—OW2—HW2B	107 (2)
C11—C12—H12A	109.5	HW3A—OW3—HW3B	106 (2)
N1—C12—H12B	109.5	HW4A—OW4—HW4B	109 (2)
C11—C12—H12B	109.5	C19—O6—H6A	111.5 (16)
H12A—C12—H12B	108.1		

Symmetry code: (i) $-x, -y+2, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6—H6A \cdots O4 ⁱ	0.92 (2)	1.47 (2)	2.392 (2)	178 (3)
N1—H1A \cdots OW1 ⁱⁱ	0.90	2.08	2.952 (2)	164
N1—H1A \cdots O6 ⁱⁱ	0.90	2.56	3.022 (2)	113
N1—H1B \cdots OW2 ⁱⁱ	0.90	1.82	2.717 (2)	176
OW1—HW1A \cdots O1 ⁱⁱⁱ	0.88 (2)	1.98 (2)	2.780 (2)	150 (2)
OW1—HW1B \cdots O3	0.85 (2)	2.38 (2)	3.041 (2)	135 (2)
OW1—HW1B \cdots O7	0.85 (2)	2.57 (2)	3.204 (2)	132 (2)
OW1—HW1B \cdots O6	0.85 (2)	2.59 (2)	3.084 (2)	118 (2)
O2—H2A \cdots O3	0.99 (2)	1.56 (2)	2.5013 (19)	159 (2)
OW2—HW2B \cdots OW4 ^{iv}	0.86 (2)	1.86 (2)	2.703 (3)	168 (3)
OW2—HW2A \cdots O5 ^{iv}	0.84 (2)	1.98 (2)	2.819 (2)	173 (3)
OW3—HW3A \cdots OW1 ^v	0.85 (2)	1.93 (2)	2.770 (2)	169 (3)
OW3—HW3B \cdots O2 ^{vi}	0.85 (2)	2.17 (2)	3.011 (3)	172 (3)
OW4—HW4A \cdots O4 ^{vii}	0.87 (2)	1.94 (2)	2.782 (2)	163 (3)
OW4—HW4B \cdots OW3	0.81 (2)	1.97 (2)	2.775 (3)	174 (3)

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x, y, z-1$; (iii) $x+1, y, z$; (iv) $-x+1, -y+2, -z+1$; (v) $-x+1, -y+1, -z+1$; (vi) $x+1, y, z-1$; (vii) $-x+1, -y+2, -z$.