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Crystal structure of 2-amino-4-methylpyridin-1-ium (2*R*,3*R*)-3-carboxy-2,3-dihydroxypropanoate monohydrate

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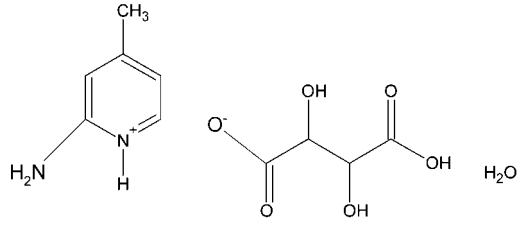
The title molecular salt, $C_6H_9N_2^+\cdot C_4H_5O_6^-\cdot H_2O$, crystallized with two 2-amino-4-methylpyridin-1-ium cations, two L-(+)-tartaric acid monoanions [systematic name: (2*R*,3*R*)-3-carboxy-2,3-dihydroxypropanoate] and two water molecules in the asymmetric unit. In the crystal, the cations, anions and water molecules are linked *via* a number of O—H···O and N—H···O hydrogen bonds, and a C—H···O hydrogen bond, forming a three-dimensional structure

Keywords: crystal structure; 2-amino-4-methylpyridin-1-ium; tartrate; L-(+)-tartaric acid.

CCDC reference: 1019274

1. Related literature

For the biological activity of pyridinium derivatives, see: Judge & Bever (2006); Schwid *et al.* (1997); Strupp *et al.* (2004). For the crystal structure of a related molecular salt involving the 2-amino-4-methylpyridin-1-ium cation, see: Hemamalini & Fun (2010).



2. Experimental

2.1. Crystal data

$C_6H_9N_2^+\cdot C_4H_5O_6^-\cdot H_2O$
M_r = 276.25
Triclinic, *P*1
a = 7.176 (3) Å
b = 9.9359 (18) Å
c = 10.716 (2) Å
α = 117.528 (5)°
β = 104.792 (7)°
γ = 91.701 (7)°
V = 645.3 (3) Å^3
Z = 2
Mo *Kα* radiation
μ = 0.12 mm^{-1}
T = 293 K
0.24 × 0.22 × 0.20 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.971, T_{max} = 0.976
10211 measured reflections
4999 independent reflections
4769 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.088
S = 1.08
4999 reflections
410 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O2	0.87 (3)	1.86 (3)	2.725 (3)	172 (3)
O1*W*—H1*WB*···O3^{i}	0.78 (3)	2.39 (4)	3.012 (3)	137 (3)
O1*W*—H1*WB*···O12	0.78 (3)	2.39 (3)	3.006 (3)	137 (3)
O3—H3···O2	0.82 (4)	1.95 (4)	2.562 (2)	131 (4)
O3—H3···O7	0.82 (4)	2.51 (4)	3.072 (3)	127 (3)
N2—H2*NA*···O1*W*^{ii}	0.84 (3)	2.00 (3)	2.819 (3)	164 (3)
N3—H3*N*···O4	0.84 (3)	2.15 (3)	2.939 (3)	157 (3)
N3—H3*N*···O6	0.84 (3)	2.44 (3)	3.047 (3)	130 (3)
N2—H2*NB*···O8^{iii}	0.83 (4)	2.41 (3)	3.032 (3)	132 (3)
N2—H2*NB*···O9^{iii}	0.83 (4)	2.21 (4)	2.942 (3)	147 (3)
O4—H4*A*···O11^{iv}	0.90 (4)	1.88 (4)	2.732 (3)	156 (3)
O2*W*—H2*WA*···O8^{i}	0.82 (4)	2.00 (4)	2.819 (3)	173 (4)
O5—H5*A*···O1^{ii}	1.16 (4)	1.41 (4)	2.534 (2)	163 (4)
N4—H4*NA*···O3	0.88 (3)	2.12 (3)	2.968 (3)	163 (3)
O7—H7*A*···O12^{ii}	1.09 (4)	1.39 (4)	2.476 (2)	174 (4)
N4—H4*NB*···O2*W*^{ii}	0.83 (4)	2.32 (4)	3.128 (4)	164 (3)
N4—H4*NB*···O12^{ii}	0.83 (4)	2.60 (4)	2.955 (4)	108 (3)
O9—H9···O2*W*	0.91 (3)	1.81 (3)	2.715 (3)	172 (3)
O10—H10*A*···O6^{v}	0.83 (3)	2.19 (3)	2.912 (3)	145 (3)
O10—H10*A*···O11	0.83 (3)	2.15 (3)	2.648 (3)	119 (3)
N1—H29···O10^{iii}	0.78 (3)	2.24 (3)	2.946 (3)	151 (3)
C5—H5···O6^{vi}	0.93	2.46	3.158 (3)	132

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

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sity of Madras, for providing data-collection and computer facilities.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2748).

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Crystal structure of 2-amino-4-methylpyridin-1-ium (2*R*,3*R*)-3-carboxy-2,3-dihydroxypropanoate monohydrate

J. V. Jovita, S. Sathya, G. Usha, R. Vasanthi and A. Ramanand

S1. Experimental

The title compound was prepared by mixing an equimolar ratio of 4-methylpyridin-2-amine (Aldrich) with L-(+)-tartaric acid (Spectrochem). When the tartaric acid was added to a saturated solution of 4-methylpyridin-2-amine in acetone a pale pink coloured precipitate was formed. The precipitate was collected and allowed to dry and powdered. Suitable crystals of the title salt were prepared by slow evaporation of a solution of the powder in methanol at room temperature.

S2. Refinement

The water, NH and OH H atoms were located from difference Fourier maps and freely refined. The C-bound H atoms were positioned geometrically and treated as riding atoms: C—H = 0.93–0.98 Å U_{iso}(H)= 1.5U_{eq}(*C*-methyl) and = 1.2U_{eq}(C) for other H atoms.

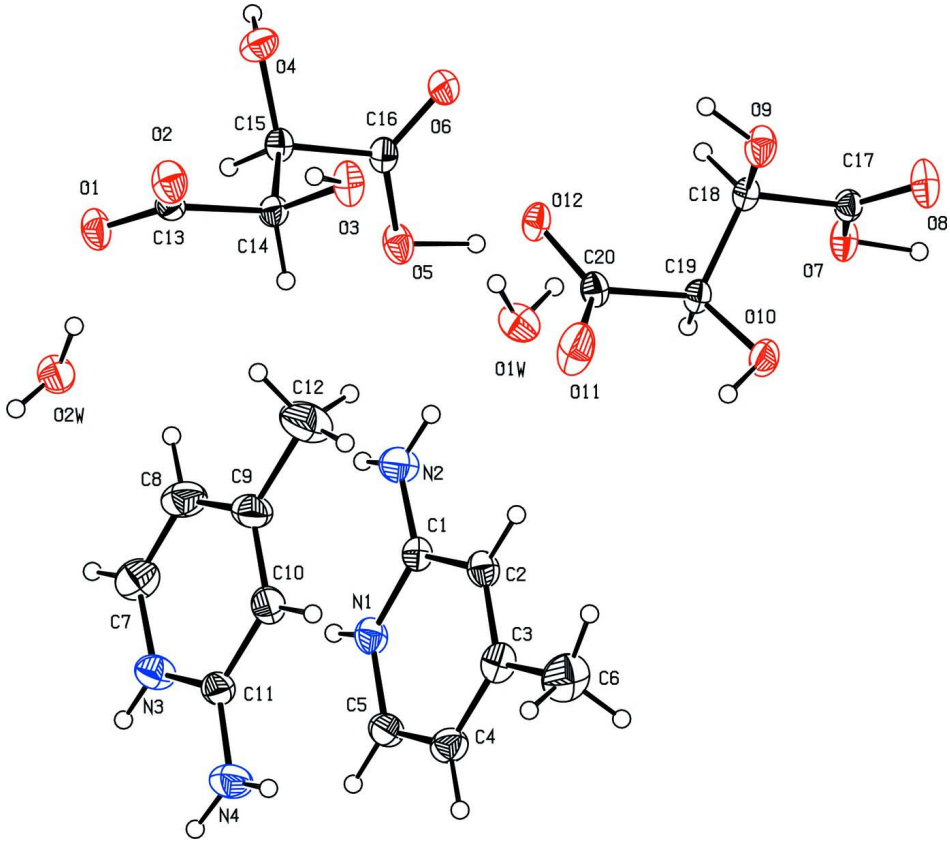

Figure 1

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

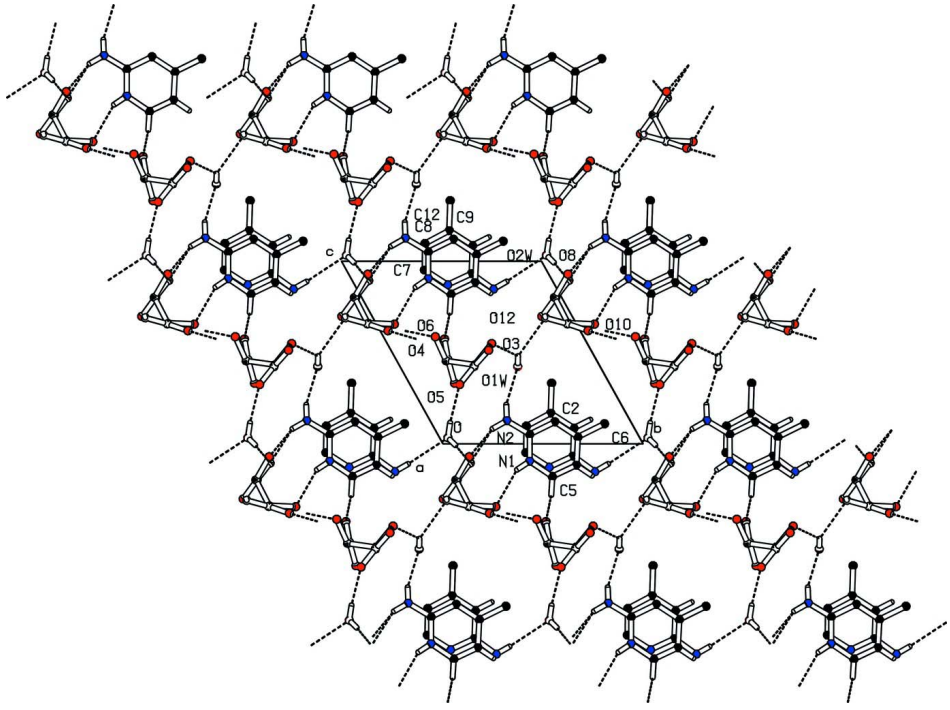

Figure 2

A view along the *a* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1 for details).

2-Amino-4-methylpyridin-1-ium (2*R*,3*R*)-3-carboxy-2,3-dihydroxypropanoate monohydrate

Crystal data

$C_6H_9N_2^+·C_4H_5O_6^-·H_2O$
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Triclinic, *P*1
Hall symbol: P 1
a = 7.176 (3) Å
b = 9.9359 (18) Å
c = 10.716 (2) Å
α = 117.528 (5)°
β = 104.792 (7)°
γ = 91.701 (7)°
V = 645.3 (3) Å^3
Z = 2
F(000) = 292
D_x = 1.422 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 8251 reflections
θ = 2.3–32.6°
μ = 0.12 mm^{-1}
T = 293 K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.971, T_{max} = 0.976
10211 measured reflections
4999 independent reflections
4769 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°, θ_{min} = 2.3°
h = −8→8
k = −12→12
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.088$
$S = 1.08$
4999 reflections
410 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
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.1123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.055 (5)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.74569 (19)	0.24776 (17)	0.32479 (15)	0.0425 (4)
O2	0.8383 (2)	0.48800 (17)	0.51261 (16)	0.0434 (4)
O3	0.4999 (2)	0.52134 (16)	0.54862 (17)	0.0421 (5)
O4	0.5543 (2)	0.25906 (18)	0.58628 (16)	0.0402 (4)
O5	0.09901 (19)	0.22642 (16)	0.33245 (15)	0.0385 (4)
O6	0.1808 (2)	0.2985 (2)	0.57363 (17)	0.0469 (5)
O7	0.73206 (18)	0.84899 (15)	0.69807 (16)	0.0366 (4)
O8	0.8096 (2)	1.08215 (17)	0.89449 (17)	0.0443 (4)
O9	1.1844 (2)	1.09612 (17)	0.92901 (15)	0.0408 (4)
O10	1.0198 (2)	1.08903 (18)	0.65507 (18)	0.0440 (5)
O11	1.3678 (3)	1.0395 (2)	0.6194 (3)	0.0676 (8)
O12	1.38559 (17)	0.86225 (14)	0.69019 (15)	0.0340 (4)
C13	0.7165 (2)	0.3699 (2)	0.42355 (19)	0.0302 (5)
C14	0.5072 (3)	0.3760 (2)	0.4327 (2)	0.0299 (5)
C15	0.4308 (2)	0.2468 (2)	0.4546 (2)	0.0295 (5)
C16	0.2212 (2)	0.25924 (19)	0.4604 (2)	0.0298 (5)
C17	0.8533 (2)	0.9703 (2)	0.80181 (19)	0.0286 (5)
C18	1.0658 (2)	0.9648 (2)	0.80165 (19)	0.0275 (5)
C19	1.0873 (2)	0.95937 (19)	0.66195 (19)	0.0290 (5)
C20	1.2990 (2)	0.9559 (2)	0.65682 (19)	0.0300 (5)
N1	0.0123 (3)	0.3863 (2)	−0.09469 (18)	0.0368 (5)
N2	0.0764 (3)	0.3843 (2)	0.1268 (2)	0.0444 (6)
C1	0.0410 (3)	0.4600 (2)	0.0521 (2)	0.0316 (5)
C2	0.0344 (3)	0.6200 (2)	0.1196 (2)	0.0355 (5)

C3	0.0001 (3)	0.6933 (2)	0.0375 (2)	0.0391 (6)
C4	−0.0325 (3)	0.6082 (3)	−0.1162 (2)	0.0417 (7)
C5	−0.0249 (3)	0.4564 (2)	−0.1787 (2)	0.0397 (6)
C6	−0.0012 (5)	0.8623 (3)	0.1096 (3)	0.0639 (9)
N3	0.4969 (3)	0.4655 (2)	0.8719 (2)	0.0450 (6)
N4	0.4515 (3)	0.6816 (3)	0.8476 (3)	0.0535 (8)
O1W	1.1106 (3)	0.58916 (19)	0.42444 (17)	0.0448 (5)
C7	0.5351 (4)	0.3913 (3)	0.9520 (3)	0.0528 (8)
C8	0.5627 (4)	0.4668 (3)	1.0974 (3)	0.0529 (8)
C9	0.5523 (3)	0.6239 (3)	1.1678 (2)	0.0499 (8)
C10	0.5161 (3)	0.6983 (3)	1.0861 (2)	0.0426 (6)
C11	0.4877 (3)	0.6164 (2)	0.9329 (2)	0.0367 (6)
C12	0.5813 (6)	0.7111 (5)	1.3315 (3)	0.0825 (13)
O2W	1.5440 (2)	1.04131 (19)	1.03282 (18)	0.0444 (5)
H3	0.613 (5)	0.565 (4)	0.575 (4)	0.067 (9)*
H4A	0.513 (5)	0.170 (4)	0.582 (3)	0.062 (8)*
H5A	−0.057 (6)	0.230 (4)	0.344 (4)	0.098 (12)*
H14	0.42260	0.36470	0.33960	0.0360*
H15	0.43150	0.14750	0.37120	0.0350*
H7A	0.582 (5)	0.862 (4)	0.698 (4)	0.082 (10)*
H9	1.300 (4)	1.068 (3)	0.959 (3)	0.055 (7)*
H10A	1.091 (4)	1.116 (3)	0.618 (3)	0.053 (7)*
H18	1.10350	0.87210	0.80480	0.0330*
H19	1.00390	0.86590	0.57650	0.0350*
H2	0.05370	0.67540	0.22070	0.0430*
H2NA	0.085 (3)	0.429 (3)	0.217 (3)	0.029 (5)*
H4	−0.05890	0.65630	−0.17380	0.0500*
H2NB	0.071 (5)	0.290 (4)	0.081 (3)	0.058 (8)*
H5	−0.04540	0.39950	−0.27990	0.0480*
H6A	0.09330	0.91180	0.08800	0.0960*
H6B	0.03140	0.90320	0.21460	0.0960*
H6C	−0.12900	0.88070	0.07290	0.0960*
H29	0.015 (4)	0.299 (3)	−0.138 (3)	0.047 (7)*
H3N	0.481 (4)	0.412 (3)	0.781 (3)	0.048 (7)*
H7	0.54230	0.28690	0.90550	0.0630*
H4NA	0.441 (4)	0.624 (3)	0.754 (3)	0.045 (7)*
H8	0.58890	0.41520	1.15170	0.0630*
H4NB	0.452 (5)	0.776 (4)	0.890 (4)	0.068 (10)*
H10	0.51030	0.80290	1.13180	0.0510*
H12A	0.70470	0.69940	1.38340	0.1240*
H12B	0.47770	0.67110	1.35330	0.1240*
H12C	0.57980	0.81830	1.36210	0.1240*
H1WA	1.033 (4)	0.554 (3)	0.457 (3)	0.052 (7)*
H1WB	1.211 (5)	0.620 (3)	0.487 (3)	0.055 (8)*
H2WA	1.617 (5)	1.059 (4)	0.992 (3)	0.062 (9)*
H2WB	1.591 (4)	1.099 (3)	1.125 (4)	0.051 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0253 (6)	0.0529 (9)	0.0359 (7)	0.0069 (6)	0.0113 (5)	0.0098 (7)
O2	0.0303 (7)	0.0451 (8)	0.0468 (8)	−0.0004 (6)	0.0125 (6)	0.0160 (7)
O3	0.0397 (8)	0.0312 (7)	0.0588 (9)	0.0085 (6)	0.0267 (7)	0.0185 (7)
O4	0.0343 (7)	0.0467 (8)	0.0487 (8)	0.0063 (6)	0.0085 (6)	0.0326 (7)
O5	0.0257 (6)	0.0419 (7)	0.0344 (7)	0.0021 (5)	0.0100 (5)	0.0076 (6)
O6	0.0457 (8)	0.0691 (10)	0.0530 (9)	0.0287 (8)	0.0309 (7)	0.0421 (8)
O7	0.0240 (6)	0.0291 (6)	0.0471 (8)	0.0044 (5)	0.0135 (5)	0.0095 (6)
O8	0.0300 (7)	0.0367 (7)	0.0509 (8)	0.0049 (6)	0.0193 (6)	0.0056 (6)
O9	0.0262 (6)	0.0410 (7)	0.0380 (7)	0.0055 (6)	0.0055 (5)	0.0076 (6)
O10	0.0455 (8)	0.0532 (9)	0.0648 (9)	0.0284 (7)	0.0330 (8)	0.0444 (8)
O11	0.0513 (9)	0.0784 (12)	0.1307 (18)	0.0294 (9)	0.0552 (11)	0.0819 (13)
O12	0.0245 (6)	0.0340 (7)	0.0484 (7)	0.0084 (5)	0.0153 (5)	0.0215 (6)
C13	0.0263 (8)	0.0413 (10)	0.0292 (8)	0.0072 (8)	0.0106 (7)	0.0209 (8)
C14	0.0258 (8)	0.0355 (9)	0.0334 (9)	0.0074 (7)	0.0111 (7)	0.0196 (7)
C15	0.0262 (8)	0.0291 (8)	0.0336 (9)	0.0057 (7)	0.0115 (7)	0.0142 (7)
C16	0.0286 (9)	0.0254 (8)	0.0402 (9)	0.0063 (7)	0.0156 (7)	0.0170 (7)
C17	0.0255 (8)	0.0306 (9)	0.0342 (9)	0.0070 (7)	0.0123 (7)	0.0176 (7)
C18	0.0233 (8)	0.0249 (8)	0.0357 (9)	0.0063 (6)	0.0106 (7)	0.0150 (7)
C19	0.0242 (8)	0.0291 (9)	0.0362 (9)	0.0073 (7)	0.0117 (7)	0.0164 (8)
C20	0.0286 (8)	0.0274 (8)	0.0360 (9)	0.0053 (7)	0.0153 (7)	0.0141 (7)
N1	0.0436 (9)	0.0281 (8)	0.0353 (8)	0.0098 (7)	0.0149 (7)	0.0109 (7)
N2	0.0646 (12)	0.0312 (9)	0.0364 (10)	0.0133 (8)	0.0164 (8)	0.0149 (8)
C1	0.0290 (8)	0.0311 (9)	0.0334 (9)	0.0063 (7)	0.0106 (7)	0.0140 (8)
C2	0.0381 (10)	0.0292 (9)	0.0329 (9)	0.0061 (7)	0.0124 (8)	0.0093 (8)
C3	0.0349 (10)	0.0307 (10)	0.0507 (11)	0.0032 (8)	0.0128 (9)	0.0193 (9)
C4	0.0407 (11)	0.0459 (12)	0.0473 (11)	0.0082 (9)	0.0135 (9)	0.0295 (10)
C5	0.0385 (10)	0.0480 (12)	0.0335 (9)	0.0073 (8)	0.0123 (8)	0.0198 (9)
C6	0.0813 (19)	0.0347 (12)	0.0725 (17)	0.0119 (12)	0.0208 (15)	0.0248 (12)
N3	0.0541 (11)	0.0444 (10)	0.0342 (9)	0.0062 (8)	0.0122 (8)	0.0179 (8)
N4	0.0698 (14)	0.0596 (13)	0.0480 (12)	0.0180 (11)	0.0234 (10)	0.0365 (11)
O1W	0.0442 (8)	0.0447 (8)	0.0366 (8)	−0.0045 (7)	0.0102 (7)	0.0143 (7)
C7	0.0550 (13)	0.0454 (12)	0.0626 (15)	0.0048 (10)	0.0142 (11)	0.0320 (12)
C8	0.0500 (13)	0.0651 (15)	0.0577 (14)	0.0048 (11)	0.0122 (11)	0.0435 (13)
C9	0.0369 (11)	0.0773 (17)	0.0395 (11)	0.0048 (11)	0.0085 (9)	0.0334 (12)
C10	0.0403 (11)	0.0472 (12)	0.0404 (10)	0.0109 (9)	0.0156 (9)	0.0194 (9)
C11	0.0319 (9)	0.0461 (11)	0.0376 (10)	0.0063 (8)	0.0126 (7)	0.0238 (9)
C12	0.092 (2)	0.108 (3)	0.0447 (14)	0.019 (2)	0.0205 (15)	0.0349 (16)
O2W	0.0345 (7)	0.0524 (9)	0.0328 (8)	0.0026 (6)	0.0055 (6)	0.0125 (7)

Geometric parameters (Å, º)

O1—C13	1.256 (2)	C2—C3	1.363 (3)
O2—C13	1.237 (3)	N2—H2NA	0.84 (3)
O3—C14	1.418 (3)	N2—H2NB	0.83 (4)
O4—C15	1.410 (2)	C3—C4	1.410 (3)

O5—C16	1.308 (2)	C3—C6	1.491 (4)
O6—C16	1.207 (2)	C4—C5	1.348 (4)
O3—H3	0.82 (4)	C2—H2	0.9300
O4—H4A	0.90 (4)	N3—C11	1.342 (3)
O5—H5A	1.16 (4)	N3—C7	1.354 (4)
O7—C17	1.285 (2)	N4—C11	1.326 (4)
O8—C17	1.216 (3)	C4—H4	0.9300
O9—C18	1.409 (2)	C5—H5	0.9300
O10—C19	1.417 (3)	C6—H6A	0.9600
O11—C20	1.216 (3)	C6—H6B	0.9600
O12—C20	1.268 (3)	C6—H6C	0.9600
C13—C14	1.531 (3)	N3—H3N	0.84 (3)
C14—C15	1.519 (3)	N4—H4NB	0.83 (4)
C15—C16	1.527 (2)	N4—H4NA	0.88 (3)
O7—H7A	1.09 (4)	C7—C8	1.335 (4)
O9—H9	0.91 (3)	C8—C9	1.399 (4)
O10—H10A	0.83 (3)	C9—C10	1.369 (4)
C14—H14	0.9800	C9—C12	1.506 (3)
C15—H15	0.9800	C10—C11	1.408 (3)
C17—C18	1.528 (2)	O1W—H1WB	0.78 (3)
C18—C19	1.520 (3)	O1W—H1WA	0.87 (3)
C19—C20	1.535 (2)	C7—H7	0.9300
N1—C5	1.355 (3)	C8—H8	0.9300
N1—C1	1.347 (3)	C10—H10	0.9300
N2—C1	1.317 (3)	C12—H12C	0.9600
C18—H18	0.9800	C12—H12A	0.9600
C19—H19	0.9800	C12—H12B	0.9600
C1—C2	1.418 (3)	O2W—H2WA	0.82 (4)
N1—H29	0.78 (3)	O2W—H2WB	0.84 (4)

C14—O3—H3	100 (3)	C1—N2—H2NB	119 (2)
C15—O4—H4A	103.0 (19)	C1—C2—C3	120.58 (17)
C16—O5—H5A	108.0 (18)	H2NA—N2—H2NB	121 (3)
O1—C13—C14	116.99 (16)	C2—C3—C4	119.4 (2)
O1—C13—O2	126.88 (16)	C4—C3—C6	120.2 (2)
O2—C13—C14	116.10 (17)	C2—C3—C6	120.37 (19)
O3—C14—C13	109.65 (16)	C3—C4—C5	119.3 (2)
O3—C14—C15	110.52 (16)	N1—C5—C4	120.27 (18)
C13—C14—C15	111.91 (17)	C1—C2—H2	120.00
C14—C15—C16	108.80 (17)	C3—C2—H2	120.00
O4—C15—C14	108.89 (16)	C7—N3—C11	122.6 (2)
O4—C15—C16	111.36 (15)	C5—C4—H4	120.00
O5—C16—O6	126.04 (16)	C3—C4—H4	120.00
O5—C16—C15	112.27 (15)	N1—C5—H5	120.00
O6—C16—C15	121.67 (16)	C4—C5—H5	120.00
C17—O7—H7A	111 (2)	C3—C6—H6B	109.00
C18—O9—H9	107.2 (19)	C3—C6—H6C	109.00
C19—O10—H10A	106 (2)	C3—C6—H6A	109.00

O3—C14—H14	108.00	H6A—C6—H6C	110.00
C13—C14—H14	108.00	H6B—C6—H6C	109.00
C15—C14—H14	108.00	H6A—C6—H6B	109.00
C16—C15—H15	109.00	C11—N3—H3N	121 (2)
O4—C15—H15	109.00	C7—N3—H3N	116 (2)
C14—C15—H15	109.00	C11—N4—H4NA	118 (2)
O7—C17—C18	114.13 (16)	H4NA—N4—H4NB	126 (3)
O7—C17—O8	125.05 (16)	C11—N4—H4NB	116 (3)
O8—C17—C18	120.83 (17)	N3—C7—C8	120.5 (3)
O9—C18—C17	108.17 (14)	C7—C8—C9	119.9 (3)
O9—C18—C19	111.32 (16)	C8—C9—C12	120.8 (3)
C17—C18—C19	109.84 (14)	C10—C9—C12	120.1 (3)
C18—C19—C20	112.12 (14)	C8—C9—C10	119.2 (2)
O10—C19—C18	108.04 (15)	C9—C10—C11	120.0 (2)
O10—C19—C20	111.07 (16)	N3—C11—C10	117.8 (2)
O11—C20—O12	125.81 (18)	N4—C11—C10	122.9 (2)
O11—C20—C19	118.3 (2)	N3—C11—N4	119.2 (2)
O12—C20—C19	115.93 (17)	H1WA—O1W—H1WB	105 (3)
C1—N1—C5	123.5 (2)	N3—C7—H7	120.00
C17—C18—H18	109.00	C8—C7—H7	120.00
C19—C18—H18	109.00	C9—C8—H8	120.00
O9—C18—H18	109.00	C7—C8—H8	120.00
C18—C19—H19	109.00	C9—C10—H10	120.00
C20—C19—H19	108.00	C11—C10—H10	120.00
O10—C19—H19	109.00	C9—C12—H12B	109.00
N1—C1—N2	120.2 (2)	C9—C12—H12C	109.00
N1—C1—C2	116.89 (19)	C9—C12—H12A	109.00
N2—C1—C2	122.93 (18)	H12A—C12—H12C	110.00
C5—N1—H29	115 (2)	H12B—C12—H12C	109.00
C1—N1—H29	122 (2)	H12A—C12—H12B	109.00
C1—N2—H2NA	120 (2)	H2WA—O2W—H2WB	109 (3)
O1—C13—C14—O3	−179.57 (18)	C18—C19—C20—O11	−135.1 (2)
O1—C13—C14—C15	57.4 (2)	C18—C19—C20—O12	46.2 (2)
O2—C13—C14—O3	−1.2 (3)	C5—N1—C1—N2	180.0 (2)
O2—C13—C14—C15	−124.3 (2)	C5—N1—C1—C2	−1.1 (3)
O3—C14—C15—O4	−63.3 (2)	C1—N1—C5—C4	0.9 (4)
O3—C14—C15—C16	58.3 (2)	N2—C1—C2—C3	179.0 (2)
C13—C14—C15—O4	59.26 (19)	N1—C1—C2—C3	0.1 (3)
C13—C14—C15—C16	−179.20 (14)	C1—C2—C3—C4	1.2 (3)
O4—C15—C16—O5	−170.89 (18)	C1—C2—C3—C6	−178.3 (2)
O4—C15—C16—O6	10.5 (3)	C6—C3—C4—C5	178.0 (2)
C14—C15—C16—O5	69.1 (2)	C2—C3—C4—C5	−1.4 (3)
C14—C15—C16—O6	−109.5 (2)	C3—C4—C5—N1	0.5 (3)
O7—C17—C18—O9	−173.74 (17)	C11—N3—C7—C8	1.0 (4)
O7—C17—C18—C19	64.6 (2)	C7—N3—C11—N4	179.3 (2)
O8—C17—C18—O9	6.1 (3)	C7—N3—C11—C10	−1.1 (4)
O8—C17—C18—C19	−115.6 (2)	N3—C7—C8—C9	−0.1 (4)

O9—C18—C19—O10	−63.25 (18)	C7—C8—C9—C12	179.5 (3)
O9—C18—C19—C20	59.5 (2)	C7—C8—C9—C10	−0.8 (4)
C17—C18—C19—O10	56.5 (2)	C8—C9—C10—C11	0.7 (3)
C17—C18—C19—C20	179.23 (17)	C12—C9—C10—C11	−179.6 (3)
O10—C19—C20—O11	−14.1 (3)	C9—C10—C11—N4	179.8 (2)
O10—C19—C20—O12	167.13 (16)	C9—C10—C11—N3	0.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O2	0.87 (3)	1.86 (3)	2.725 (3)	172 (3)
O1*W*—H1*WB*···O3^{i}	0.78 (3)	2.39 (4)	3.012 (3)	137 (3)
O1*W*—H1*WB*···O12	0.78 (3)	2.39 (3)	3.006 (3)	137 (3)
O3—H3···O2	0.82 (4)	1.95 (4)	2.562 (2)	131 (4)
O3—H3···O7	0.82 (4)	2.51 (4)	3.072 (3)	127 (3)
N2—H2*NA*···O1*W*^{ii}	0.84 (3)	2.00 (3)	2.819 (3)	164 (3)
N3—H3*N*···O4	0.84 (3)	2.15 (3)	2.939 (3)	157 (3)
N3—H3*N*···O6	0.84 (3)	2.44 (3)	3.047 (3)	130 (3)
N2—H2*NB*···O8^{iii}	0.83 (4)	2.41 (3)	3.032 (3)	132 (3)
N2—H2*NB*···O9^{iii}	0.83 (4)	2.21 (4)	2.942 (3)	147 (3)
O4—H4*A*···O11^{iv}	0.90 (4)	1.88 (4)	2.732 (3)	156 (3)
O2*W*—H2*WA*···O8^{i}	0.82 (4)	2.00 (4)	2.819 (3)	173 (4)
O5—H5*A*···O1^{ii}	1.16 (4)	1.41 (4)	2.534 (2)	163 (4)
N4—H4*NA*···O3	0.88 (3)	2.12 (3)	2.968 (3)	163 (3)
O7—H7*A*···O12^{ii}	1.09 (4)	1.39 (4)	2.476 (2)	174 (4)
N4—H4*NB*···O2*W*^{ii}	0.83 (4)	2.32 (4)	3.128 (4)	164 (3)
N4—H4*NB*···O12^{ii}	0.83 (4)	2.60 (4)	2.955 (4)	108 (3)
O9—H9···O2*W*	0.91 (3)	1.81 (3)	2.715 (3)	172 (3)
O10—H10*A*···O6^{v}	0.83 (3)	2.19 (3)	2.912 (3)	145 (3)
O10—H10*A*···O11	0.83 (3)	2.15 (3)	2.648 (3)	119 (3)
N1—H29···O10^{iii}	0.78 (3)	2.24 (3)	2.946 (3)	151 (3)
C5—H5···O6^{vi}	0.93	2.46	3.158 (3)	132

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