Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Aminopyrimidinium dihydrogen phosphate monohydrate

Houda Marouani,[a]* Salem S. Al-Deyab[b] and Mohamed Rzaigui[a]

[a]Laboratoire de Chimie des Matériaux, Faculté des Sciences de Bizerte, 7021 Zarzouna Bizerte, Tunisia, and [b]Petrochemical Research Chair, College of Science, King Saud University, Riadh, Saudi Arabia
Correspondence e-mail: houda.marouani@fsb.rnu.tn

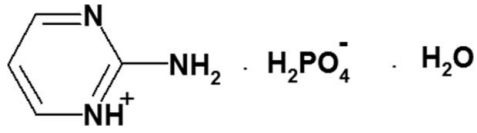
Received 16 March 2011; accepted 22 March 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.124; data-to-parameter ratio = 27.7.

In the title compound, $C_4H_6N_3^+\cdot H_2O_4P^-\cdot H_2O$, the pyrimidinium ring is essentially planar, with an r.m.s. deviation of 0.0016 Å. In the structure, pairs of symmetry-related anions are connected into centrosymmetric clusters *via* strong O—H···O hydrogen bonds forming six-membered rings with an $R_2^2(6)$ motif. These clusters are interconnected *via* water molecules through O*W*—H···O hydrogen bonds, building an infinite layer parallel to the *ab* plane. Moreover, infinite chains of 2-aminopyrimidinium cations spread along the *a*-axis direction. These chains are connected to the inorganic layer through N—H···O, C—H···O and C—H···N hydrogen bonds, which, together with electrostatic and van der Waals interactions, contribute to the cohesion and stability of the network in the crystal structure.

Related literature

For the biological activity of aminopyrimidinium derivatives, see: Grier *et al.* (1980); Gueiffier *et al.* (1996); Rival *et al.* (1991); Li *et al.* (2009). For related structures, see: Cheng *et al.* (2010); Narayana *et al.* (2008). For graph-set notation of hydrogen bonding, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_4H_6N_3^+\cdot H_2PO_4^-\cdot H_2O$
M_r = 211.12
Triclinic, $P\overline{1}$
a = 6.212 (3) Å
b = 8.600 (4) Å
c = 9.462 (2) Å
α = 109.56 (3)°
β = 106.38 (2)°
γ = 95.50 (2)°
V = 446.7 (3) Å^3
Z = 2
Ag $K\alpha$ radiation
λ = 0.56083 Å
μ = 0.17 mm^{-1}
T = 293 K
0.40 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
6572 measured reflections
4373 independent reflections
3189 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
2 standard reflections every 120 min
intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.124
S = 1.03
4373 reflections
158 parameters
3 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O4^{i}	0.83 (3)	1.80 (3)	2.6372 (18)	179 (2)
O2—H2*O*···O*W*	0.82 (3)	1.80 (3)	2.6132 (18)	175 (2)
O*W*—H1*W*···O3^{ii}	0.84 (1)	1.96 (1)	2.7882 (17)	166 (2)
O*W*—H2*W*···O4^{iii}	0.83 (1)	1.95 (1)	2.7843 (16)	177 (2)
N1—H1···O4^{iv}	0.83 (2)	2.08 (2)	2.9070 (18)	177 (2)
N1—H2···O3^{v}	0.89 (2)	2.00 (2)	2.873 (2)	166 (2)
N2—H3···O3^{iv}	0.87 (2)	1.78 (2)	2.6535 (16)	175 (2)
C2—H4···N3^{iii}	0.91 (2)	2.62 (2)	3.513 (2)	169 (2)
C3—H5···O*W*	0.95 (2)	2.56 (2)	3.477 (2)	164 (2)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We would like to acknowledge the support provided by the Secretary of State for Scientific Research and Technology of Tunisia.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2399).

References

Bernstein, J., David, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Cheng, X.-L., Gao, S. & Ng, S. W. (2010). *Acta Cryst.* E**66**, o127.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Grier, N., Harris, E. H., Joshua, H., Patchett, A. A., Witzel, B. E. & Dybas, R. A. (1980). *J. Coat. Technol.* A**52**, 57–63.
Gueiffier, A., Lhassani, M., Elhakmaoui, A., Snoeck, R., Andrei, G., Chavignon, O., Teulade, J.-C., Kerbal, A., Essassi, E. M., Debouzy, J.-C., Witvrouw, M., Blache, Y., De Balzarini, J., Clercq, E. & Chapat, J.-P. (1996). *J. Med. Chem.* **39**, 2856–2859.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Li, H.-Y., Brooks, H. B., Crich, J. Z., Henry, J. R., Sawyer, J. S. & Wang, Y. (2009). Eur. Patent (Eli Lilly & Company).
Narayana, B., Sarojini, B. K., Prakash Kamath, K., Yathirajan, H. S. & Bolte, M. (2008). *Acta Cryst.* E**64**, o117–o118.
Rival, Y., Grassy, G., Taudou, A. & Ecalle, R. (1991). *Eur. J. Med. Chem.* **26**, 13–18.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o970–o971 [doi:10.1107/S1600536811010658]

2-Aminopyrimidinium dihydrogen phosphate monohydrate

Houda Marouani, Salem S. Al-Deyab and Mohamed Rzaigui

S1. Comment

Aminopyrimidinium salts are highly active antimicrobial agents with a low acute mammalian toxicity (Grier *et al.*, 1980). In addition, pyrimidine derivatives possess considerable biological activity and have been widely used in medicinal applications as antiviral agents (Gueiffier *et al.*, 1996), antifungal agents (Rival *et al.*, 1991). Moreover, imidazolidinonyl aminopyrimidine compounds have been investigated for the treatment of cancer (Li *et al.*, 2009). In order to search for new materials for these applications, we have attempted to combine pyrimidine derivatives with phosphate species. In this paper we report the preparation and the crystal structure of the title compound, (I).

The asymmetric unit of (I) contains a $H_2PO_4^-$ anion, a 2-aminopyrimidinium cation and a water molecule (Fig. 1). The pyrimidinium ring is essentially planar with an rms deviation of 0.0016 Å. The interatomic bond lengths and angles in (I) do not show significant deviation from those reported in related 2-aminopyrimidinium salts (Narayana, *et al.*, 2008; Cheng, *et al.*, 2010).

In the structure, pairs of symmetry-related anions are connected into centrosymmetric clusters *via* strong O—H···O hydrogen bonds forming six-membred rings which may be described as $R_2^2(6)$ motif in the graph-set notation (Bernstein *et al.*, 1995). These clusters are interconnected *via* water molecules through OW—H···O hydrogen bonds to build an infinite layer parallel to the *ab* plane. Moreover, infinite chains of 2-aminopyrimidinium cations spread along the *a* direction. These chains are connected to the inorganic layer through H-bonds: N—H···O, C—H···O and C—H···N (Tab. 1 & Fig. 2).

S2. Experimental

A small quantity of H_3PO_4 (3 mmol) was added dropwise to a solution of 2-aminopyrimidine (3 mmol in 20 ml water). A precipitate was formed which was dissolved in water (20 ml) and the solution was allowed to evaporate slowly at room temperature until the formation of colorless prismatic crystals with dimensions suitable for a crystallographic study.

S3. Refinement

All H atoms were located in difference Fourier maps and were allowed to refine with isotropic displacement parameters U_{iso}. The H-atoms of water molecule were refined with a distance restraint of O—H = 0.85 (1) Å.

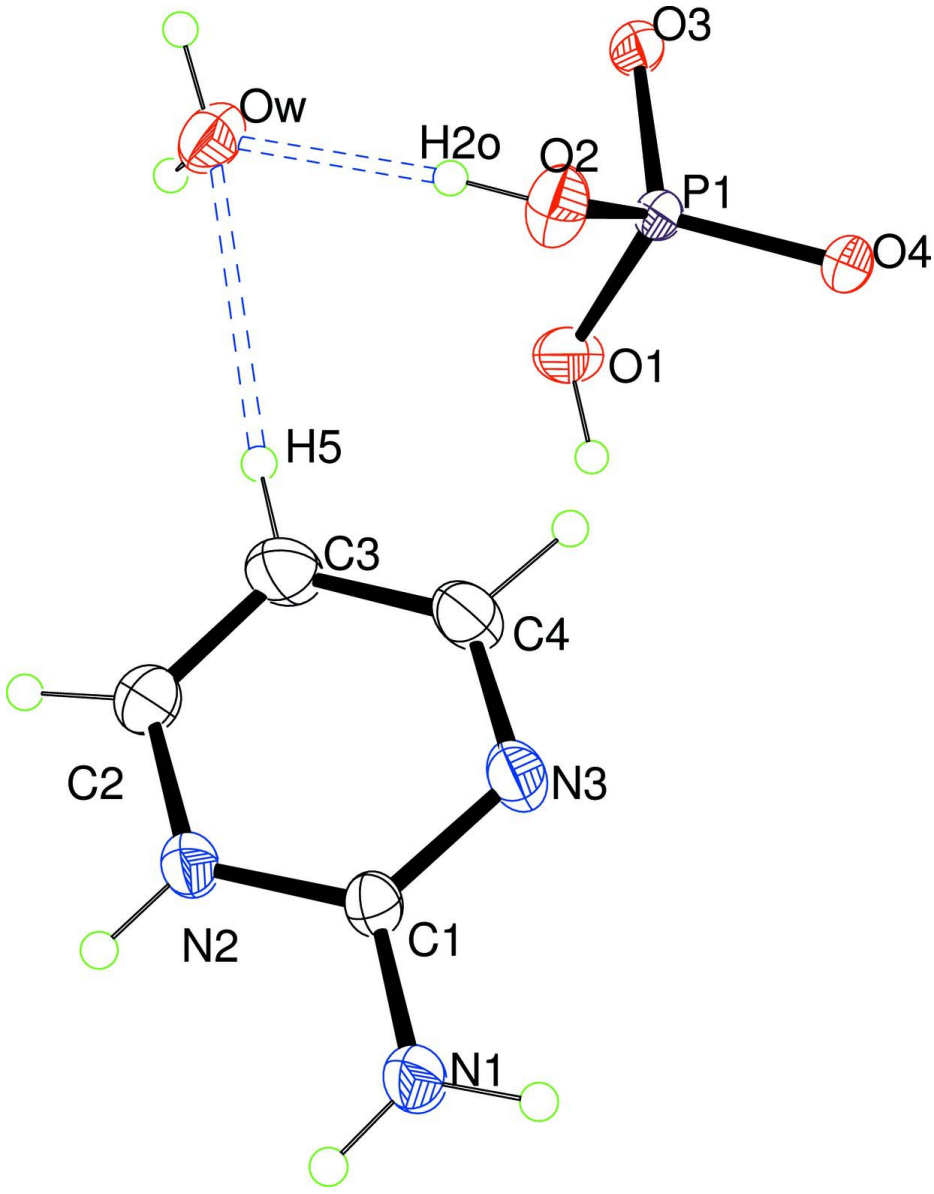


Figure 1

An *ORTEP* view of of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by small spheres of arbitrary radii.

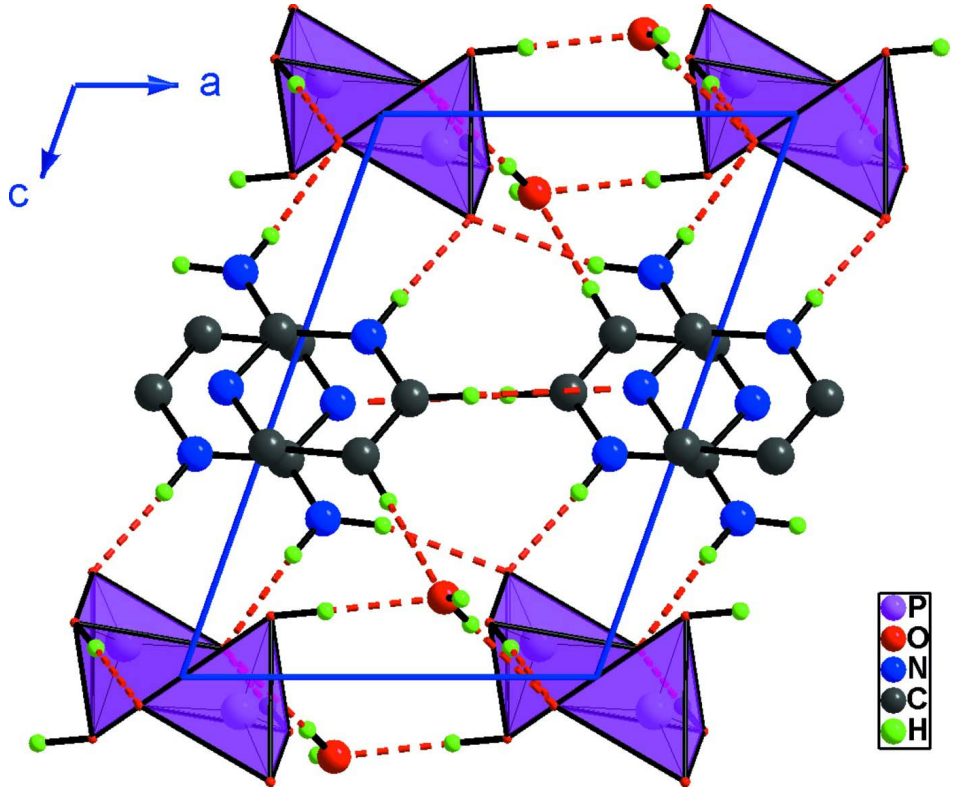


Figure 2

Projection of (I) along the *b* axis.

2-Aminopyrimidinium dihydrogen phosphate monohydrate

Crystal data

$C_4H_6N_3^+·H_2PO_4^-·H_2O$
$M_r = 211.12$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.212$ (3) Å
$b = 8.600$ (4) Å
$c = 9.462$ (2) Å
$\alpha = 109.56$ (3)°
$\beta = 106.38$ (2)°
$\gamma = 95.50$ (2)°
$V = 446.7$ (3) Å^3
$Z = 2$
$F(000) = 220$
$D_x = 1.570$ Mg m^{-3}
Ag $K\alpha$ radiation, $\lambda = 0.56083$ Å
Cell parameters from 25 reflections
$\theta = 9$–11°
$\mu = 0.17$ mm^{-1}
$T = 293$ K
Prism, colorless
0.40 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
6572 measured reflections
4373 independent reflections
3189 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.0°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -5 \rightarrow 15$
2 standard reflections every 120 min
intensity decay: 3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.124$
$S = 1.03$
4373 reflections
158 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.0087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.16874 (4)	0.74641 (3)	1.06016 (4)	0.02665 (8)
O3	0.30367 (14)	0.91480 (10)	1.18597 (11)	0.03404 (18)
O4	−0.07714 (13)	0.70387 (10)	1.05045 (11)	0.03351 (18)
O1	0.30374 (15)	0.61277 (12)	1.09639 (14)	0.0442 (3)
O2	0.16298 (17)	0.73861 (14)	0.89151 (12)	0.0433 (2)
OW	0.56449 (16)	0.79100 (13)	0.85902 (13)	0.0406 (2)
N2	0.15897 (16)	0.12980 (13)	0.39597 (13)	0.03242 (19)
N3	−0.14127 (17)	0.21993 (14)	0.48619 (13)	0.0348 (2)
N1	−0.20502 (17)	−0.03637 (13)	0.28151 (13)	0.0352 (2)
C1	−0.06315 (18)	0.10520 (14)	0.38722 (13)	0.02783 (19)
C2	0.3116 (2)	0.27019 (17)	0.50499 (18)	0.0419 (3)
C4	0.0099 (2)	0.35703 (17)	0.59250 (17)	0.0401 (3)
C3	0.2421 (3)	0.38927 (18)	0.60766 (19)	0.0467 (3)
H1W	0.608 (3)	0.8881 (14)	0.862 (2)	0.052 (5)*
H1	−0.164 (3)	−0.111 (2)	0.218 (2)	0.049 (5)*
H1O	0.232 (4)	0.513 (3)	1.051 (3)	0.075 (7)*
H3	0.201 (3)	0.054 (2)	0.327 (2)	0.049 (5)*
H6	−0.051 (3)	0.439 (2)	0.662 (2)	0.044 (4)*
H5	0.347 (4)	0.487 (3)	0.689 (3)	0.064 (6)*
H2	−0.355 (4)	−0.047 (2)	0.269 (2)	0.053 (5)*
H4	0.455 (4)	0.272 (3)	0.499 (2)	0.060 (6)*
H2W	0.668 (3)	0.761 (2)	0.916 (2)	0.065 (6)*
H2O	0.289 (4)	0.761 (3)	0.883 (3)	0.078 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01938 (11)	0.02235 (12)	0.03390 (15)	0.00234 (8)	0.00888 (9)	0.00599 (9)
O3	0.0271 (3)	0.0257 (3)	0.0406 (5)	−0.0005 (3)	0.0135 (3)	0.0018 (3)
O4	0.0216 (3)	0.0284 (3)	0.0472 (5)	0.0027 (3)	0.0124 (3)	0.0102 (3)
O1	0.0261 (4)	0.0291 (4)	0.0649 (7)	0.0059 (3)	0.0023 (4)	0.0132 (4)
O2	0.0328 (4)	0.0556 (6)	0.0366 (5)	0.0013 (4)	0.0123 (4)	0.0130 (4)
OW	0.0297 (4)	0.0428 (5)	0.0520 (6)	0.0031 (3)	0.0113 (4)	0.0244 (4)
N2	0.0256 (4)	0.0323 (4)	0.0358 (5)	0.0064 (3)	0.0124 (4)	0.0068 (4)
N3	0.0295 (4)	0.0376 (5)	0.0353 (5)	0.0116 (4)	0.0132 (4)	0.0080 (4)
N1	0.0256 (4)	0.0340 (5)	0.0374 (5)	0.0050 (3)	0.0100 (4)	0.0038 (4)
C1	0.0244 (4)	0.0311 (4)	0.0284 (5)	0.0080 (3)	0.0093 (3)	0.0108 (4)
C2	0.0274 (5)	0.0376 (6)	0.0513 (8)	0.0022 (4)	0.0116 (5)	0.0079 (5)
C4	0.0398 (6)	0.0363 (5)	0.0388 (6)	0.0132 (5)	0.0132 (5)	0.0061 (5)
C3	0.0373 (6)	0.0356 (6)	0.0484 (8)	0.0031 (5)	0.0081 (5)	−0.0006 (5)

Geometric parameters (Å, º)

P1—O4	1.5055 (11)	N3—C4	1.3207 (18)
P1—O3	1.5056 (12)	N3—C1	1.3473 (15)
P1—O1	1.5583 (11)	N1—C1	1.3194 (16)
P1—O2	1.5645 (11)	N1—H1	0.83 (2)
O1—H1O	0.83 (3)	N1—H2	0.89 (2)
O2—H2O	0.82 (3)	C2—C3	1.354 (2)
OW—H1W	0.843 (9)	C2—H4	0.91 (2)
OW—H2W	0.834 (9)	C4—C3	1.399 (2)
N2—C2	1.3473 (17)	C4—H6	0.98 (2)
N2—C1	1.3503 (15)	C3—H5	0.95 (2)
N2—H3	0.87 (2)		
O4—P1—O3	114.86 (5)	C1—N1—H2	118.5 (12)
O4—P1—O1	111.37 (6)	H1—N1—H2	118.3 (17)
O3—P1—O1	106.04 (6)	N1—C1—N3	119.50 (10)
O4—P1—O2	106.56 (6)	N1—C1—N2	118.97 (10)
O3—P1—O2	110.54 (6)	N3—C1—N2	121.51 (10)
O1—P1—O2	107.27 (7)	N2—C2—C3	119.76 (12)
P1—O1—H1O	115.6 (18)	N2—C2—H4	112.8 (13)
P1—O2—H2O	115.0 (18)	C3—C2—H4	127.4 (13)
H1W—OW—H2W	112.2 (18)	N3—C4—C3	124.26 (12)
C2—N2—C1	121.10 (11)	N3—C4—H6	115.4 (10)
C2—N2—H3	120.4 (13)	C3—C4—H6	120.3 (10)
C1—N2—H3	118.5 (13)	C2—C3—C4	116.54 (13)
C4—N3—C1	116.83 (11)	C2—C3—H5	121.6 (14)
C1—N1—H1	122.7 (13)	C4—C3—H5	121.8 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O4^{i}	0.83 (3)	1.80 (3)	2.6372 (18)	179 (2)
O2—H2*O*···O*W*	0.82 (3)	1.80 (3)	2.6132 (18)	175 (2)
O*W*—H1*W*···O3^{ii}	0.84 (1)	1.96 (1)	2.7882 (17)	166 (2)
O*W*—H2*W*···O4^{iii}	0.83 (1)	1.95 (1)	2.7843 (16)	177 (2)
N1—H1···O4^{iv}	0.83 (2)	2.08 (2)	2.9070 (18)	177 (2)
N1—H2···O3^{v}	0.89 (2)	2.00 (2)	2.873 (2)	166 (2)
N2—H3···O3^{iv}	0.87 (2)	1.78 (2)	2.6535 (16)	175 (2)
C2—H4···N3^{iii}	0.91 (2)	2.62 (2)	3.513 (2)	169 (2)
C3—H5···O*W*	0.95 (2)	2.56 (2)	3.477 (2)	164 (2)

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