Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3-hydroxymethyl-2-methylenecyclopentyl]-1,9-dihydro-6*H*-purin-6-one monohydrate

Bin Jiang[a] and Zhilu Liu[b]*

[a]Department of Pharmacy, Shandong Medical College, Jinan 250002, People's Republic of China, and [b]State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 73000, People's Republic of China
Correspondence e-mail: liuzhilu2009@yahoo.com.cn

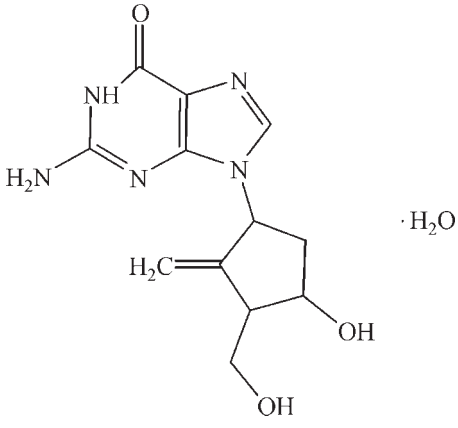
Received 4 August 2009; accepted 19 August 2009

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.079; data-to-parameter ratio = 6.8.

In the crystal of the title compound, $C_{12}H_{15}N_5O_3\cdot H_2O$, the component species are linked by N—H···N, N—H···O, O—H···N and O—H···O hydrogen bonds, forming a three-dimesnional network.

Related literature

For background, see: Czarnik (2008).



Experimental

Crystal data

$C_{12}H_{15}N_5O_3\cdot H_2O$
M_r = 295.31
Orthorhombic, $C222_1$
a = 6.9986 (10) Å
b = 11.6229 (10) Å
c = 33.932 (3) Å
V = 2760.1 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 273 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.987, T_{max} = 0.991
6725 measured reflections
1377 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.079
S = 1.00
1377 reflections
204 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 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
O1—H1···N4^{i}	0.82	2.04	2.857 (2)	172
O2—H2A···O1W^{ii}	0.82	1.83	2.639 (3)	169
N3—H3B···O3^{iii}	0.86	2.24	3.039 (3)	154
N5—H5C···N2^{iii}	0.97 (3)	1.86 (3)	2.829 (3)	177 (3)
O1W—H2W···O1^{iv}	0.819 (19)	2.113 (10)	2.900 (3)	161 (3)
O1W—H1W···O2^{v}	0.821 (12)	2.000 (16)	2.783 (3)	159 (4)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; 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*.

This work was supported by the Chinese Academy of Sciences ('Hundred Talents Program') and the Ministry of Science and Technology of China (project of '973' plan, No. 2007CB607606).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5032).

References

Bruker (2004). *APEX2*, *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Czarnik, A. W. (2008). *J. Comb. Chem.* **10**, 1–2.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2232 [doi:10.1107/S1600536809032966]

2-Amino-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3-hydroxymethyl-2-methylenecyclopentyl]-1,9-dihydro-6*H*-purin-6-one monohydrate

Bin Jiang and Zhilu Liu

S1. Comment

The research of anti-hepatitis B virus (anti-HBV) drug has long been one of the serious diseases threatening human's health, and thus searching for effective medicines to cure such illness has led to significant interest over the past decades (Czarnik, 2008). In this article, we report the crystal structural characterization of 2-Amino-1,9-dihydro-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3- (hydroxymethyl)-2-methylenecyclopentyl]-6*H*-purin-6-one.

As shown in figure 1, the asymmetrical unit contains one 2-Amino-1,9-dihydro-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3-(hydroxymethyl)-2 -methylenecyclopentyl]-6*H*-purin-6-one and one water molecule. In addition, it is noteworthy that the multipoint hydrogen-bonding links also exist between the hydrogen atoms including N3—H3B···O3, 3.039 (3) Å; O1—H1···N4, 2.861 (2) Å; O2—H2A···O1W, 2.634 (2) Å; O1W—H2W···O1, 2.899 (3) Å; O1W—H1W···O2, 2.786 (3) Å; this may make a contribution to stabilizing the chain structure, shown in figure 2.

S2. Experimental

The reaction was performed in a 25-ml Teflon-lined stainless steel vessel. The powder of 2-amino-1,9-dihydro-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3-(hydroxymethyl)-2 -methylenecyclopentyl]-6*H*-purin-6-one (1 mmol) in 5 ml water and 5 ml etanol was heated to 443 K and kept at this temperature for one day. Upon cooling, colourless blocks of (I) were recovered. Anal. Calc. for $C_{12}H_{17}N_5O_4$: C 48.76, H 5.08, N 23.70%; Found: 48.68, H 5.05, N 23.66%.

S3. Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement.

All hydrogen atoms bound to carbon were refined using a riding model with C—H = 0.93 (aryl), 0.97 (methylene) or 0.96 Å (methyl), and with U_{iso}(H) = 1.2U_{eq}(C) (aryl, methylene) or 1.5U_{eq}(C) (methyl). The water H atoms were refined with restraints of O—H = 0.82 (1)Å and H···H = 1.38 (1)Å.

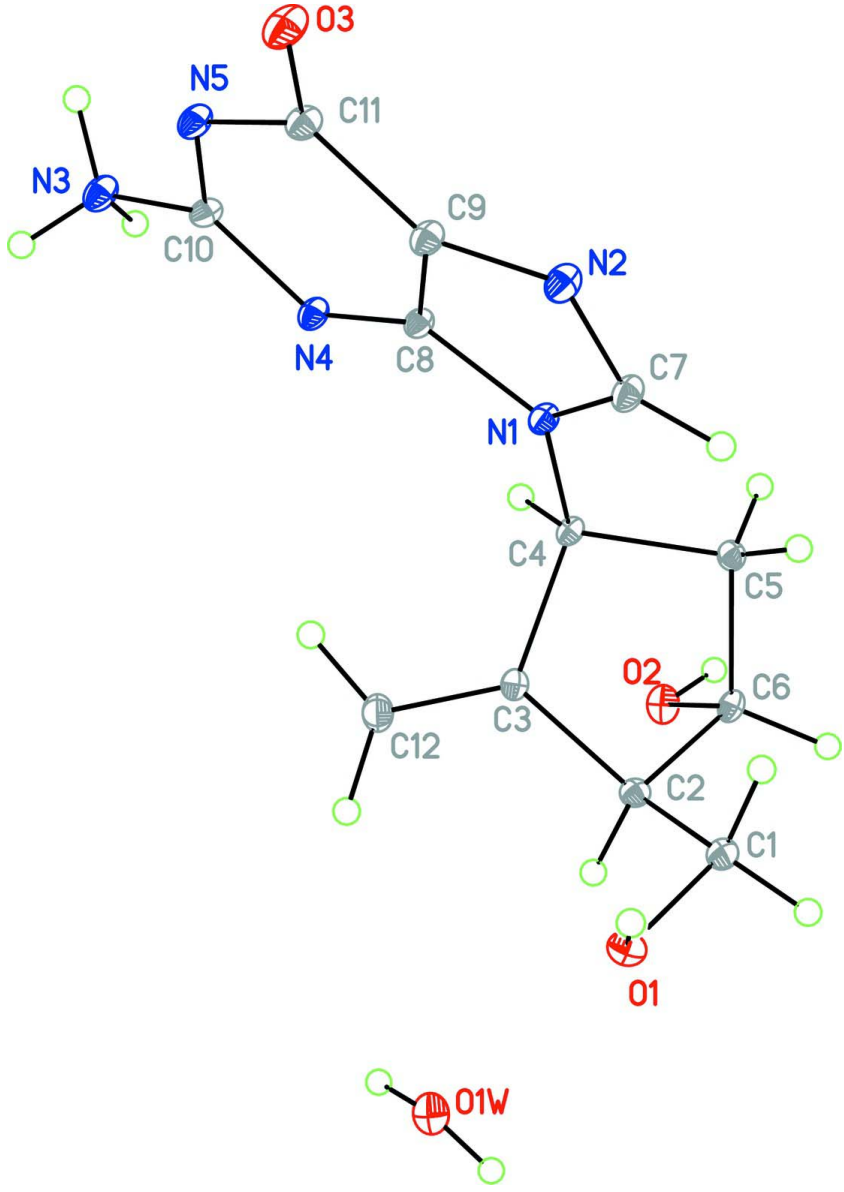


Figure 1

A view of (I) with displacement ellipsoids drawn at the 30% probability level.

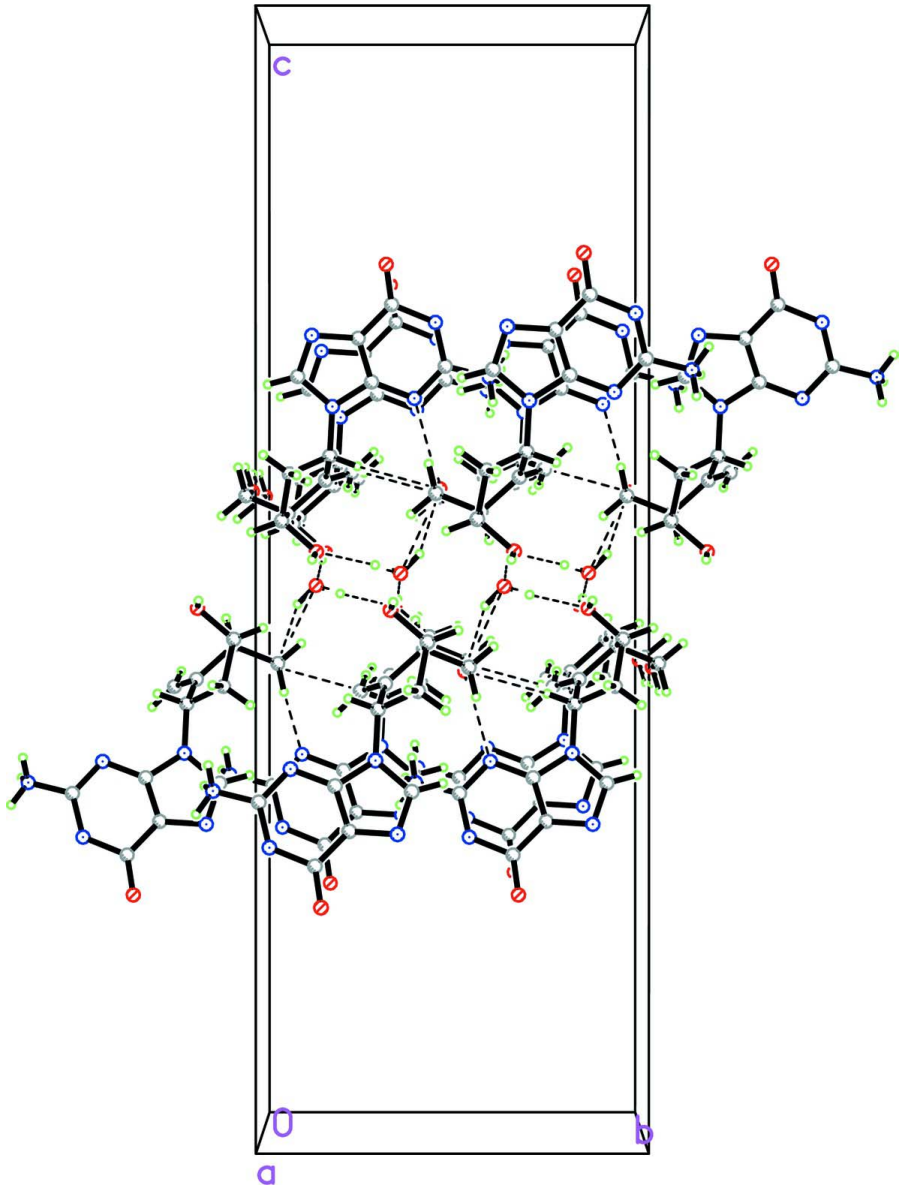


Figure 2

A view of (I) packing strcuture.

2-Amino-9-[(1*S*,3*R*,4*S*)-4-hydroxy-3-hydroxymethyl- 2-methylenecyclopentyl]-1,9-dihydro-6*H*-purin-6-one monohydrate

Crystal data

$C_{12}H_{15}N_5O_3 \cdot H_2O$
$M_r = 295.31$
Orthorhombic, $C222_1$
Hall symbol: C 2c 2
$a = 6.9986\ (10)$ Å
$b = 11.6229\ (10)$ Å
$c = 33.932\ (3)$ Å
$V = 2760.1\ (5)$ Å^3
$Z = 8$
$F(000) = 1248$
$D_x = 1.421$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1377 reflections
$\theta = 3.4–25.0°$
$\mu = 0.11$ mm^{-1}
$T = 273$ K
Block, colorless
0.12 × 0.10 × 0.08 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, 2004)
T_{min} = 0.987, T_{max} = 0.991
6725 measured reflections
1377 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°, θ_{min} = 3.4°
h = −8→8
k = −11→13
l = −33→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.079
S = 1.00
1377 reflections
204 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.4836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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}
C1	0.5675 (3)	−0.0420 (2)	0.57539 (7)	0.0345 (5)
H1A	0.5960	−0.1042	0.5573	0.041*
H1B	0.6267	−0.0597	0.6005	0.041*
C2	0.6493 (3)	0.0717 (2)	0.55918 (6)	0.0297 (5)
H2	0.5838	0.0930	0.5347	0.036*
C3	0.6333 (3)	0.16870 (18)	0.58921 (6)	0.0269 (5)
C4	0.8242 (3)	0.18411 (18)	0.60945 (6)	0.0267 (5)
H4	0.8800	0.2562	0.5999	0.032*
C5	0.9461 (3)	0.0841 (2)	0.59303 (7)	0.0314 (5)
H5A	1.0796	0.1060	0.5911	0.038*
H5B	0.9355	0.0164	0.6096	0.038*
C6	0.8611 (3)	0.0616 (2)	0.55207 (6)	0.0306 (5)
H6	0.8961	−0.0146	0.5420	0.037*
C7	0.7957 (4)	0.10135 (19)	0.67986 (7)	0.0364 (6)
H7	0.7995	0.0242	0.6727	0.044*

C8	0.8003 (3)	0.29145 (18)	0.67510 (6)	0.0293 (5)
C9	0.7746 (4)	0.25703 (19)	0.71395 (7)	0.0338 (5)
C10	0.7892 (3)	0.48170 (19)	0.68899 (6)	0.0327 (5)
C11	0.7511 (4)	0.34291 (19)	0.74423 (7)	0.0353 (5)
C12	0.4843 (4)	0.2341 (2)	0.59666 (9)	0.0445 (6)
H12A	0.4935	0.2931	0.6151	0.053*
H12B	0.3697	0.2215	0.5835	0.053*
N1	0.8142 (3)	0.19086 (16)	0.65304 (5)	0.0310 (4)
N2	0.7723 (3)	0.13654 (16)	0.71662 (6)	0.0395 (5)
N3	0.7949 (3)	0.59520 (16)	0.67918 (6)	0.0424 (5)
H3A	0.8106	0.6153	0.6550	0.051*
H3B	0.7829	0.6468	0.6972	0.051*
N4	0.8083 (3)	0.40206 (16)	0.66034 (5)	0.0331 (5)
N5	0.7614 (3)	0.45538 (16)	0.72814 (5)	0.0362 (5)
O1	0.3698 (2)	−0.03239 (15)	0.58020 (5)	0.0387 (4)
H1	0.3405	−0.0525	0.6026	0.058*
O2	0.9107 (3)	0.15191 (15)	0.52463 (5)	0.0422 (5)
H2A	1.0233	0.1455	0.5183	0.063*
O3	0.7248 (3)	0.32814 (15)	0.78032 (5)	0.0499 (5)
O1W	0.2565 (3)	0.13435 (18)	0.49421 (6)	0.0500 (5)
H2W	0.274 (5)	0.0919 (17)	0.4753 (5)	0.069 (12)*
H1W	0.274 (7)	0.2029 (6)	0.4897 (8)	0.102 (16)*
H5C	0.754 (5)	0.518 (2)	0.7470 (8)	0.080 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0340 (12)	0.0315 (13)	0.0380 (13)	−0.0029 (10)	−0.0012 (10)	−0.0008 (11)
C2	0.0331 (12)	0.0313 (12)	0.0246 (10)	−0.0001 (10)	−0.0015 (9)	0.0007 (9)
C3	0.0304 (11)	0.0236 (11)	0.0269 (10)	0.0008 (9)	0.0050 (9)	0.0052 (9)
C4	0.0325 (11)	0.0223 (11)	0.0252 (10)	−0.0022 (9)	0.0036 (8)	−0.0009 (9)
C5	0.0267 (11)	0.0321 (12)	0.0355 (12)	0.0022 (10)	0.0017 (9)	−0.0033 (10)
C6	0.0371 (12)	0.0267 (11)	0.0282 (11)	0.0004 (10)	0.0072 (9)	−0.0033 (9)
C7	0.0539 (15)	0.0242 (11)	0.0312 (12)	0.0028 (11)	0.0004 (11)	0.0012 (9)
C8	0.0357 (12)	0.0269 (11)	0.0255 (11)	−0.0007 (10)	0.0004 (9)	−0.0028 (9)
C9	0.0461 (13)	0.0291 (11)	0.0260 (11)	−0.0006 (11)	0.0023 (11)	0.0004 (9)
C10	0.0390 (13)	0.0317 (12)	0.0272 (11)	−0.0021 (11)	0.0011 (10)	−0.0027 (9)
C11	0.0455 (13)	0.0346 (11)	0.0258 (12)	−0.0019 (12)	0.0012 (10)	0.0007 (9)
C12	0.0395 (14)	0.0359 (14)	0.0580 (17)	0.0044 (12)	0.0021 (12)	−0.0019 (12)
N1	0.0416 (11)	0.0256 (9)	0.0260 (9)	−0.0015 (9)	0.0005 (8)	−0.0019 (8)
N2	0.0594 (13)	0.0299 (10)	0.0292 (10)	−0.0009 (10)	0.0029 (10)	0.0047 (8)
N3	0.0707 (15)	0.0281 (10)	0.0284 (10)	−0.0006 (11)	0.0061 (10)	−0.0004 (8)
N4	0.0477 (12)	0.0273 (10)	0.0243 (9)	−0.0005 (9)	0.0033 (9)	−0.0017 (8)
N5	0.0544 (12)	0.0298 (10)	0.0242 (9)	−0.0007 (10)	0.0036 (9)	−0.0041 (8)
O1	0.0332 (9)	0.0438 (10)	0.0389 (9)	−0.0070 (8)	0.0015 (7)	0.0058 (8)
O2	0.0431 (10)	0.0434 (10)	0.0401 (9)	0.0010 (8)	0.0184 (8)	0.0074 (8)
O3	0.0824 (14)	0.0433 (10)	0.0239 (8)	−0.0004 (10)	0.0077 (8)	0.0016 (7)
O1W	0.0490 (11)	0.0500 (12)	0.0512 (12)	0.0011 (11)	0.0141 (9)	0.0074 (10)

Geometric parameters (Å, º)

C1—O1	1.398 (3)	C8—N4	1.381 (3)
C1—C2	1.542 (3)	C8—C9	1.389 (3)
C1—H1A	0.9700	C8—N1	1.392 (3)
C1—H1B	0.9700	C9—N2	1.403 (3)
C2—C6	1.506 (3)	C9—C11	1.442 (3)
C2—C3	1.524 (3)	C10—N4	1.349 (3)
C2—H2	0.9800	C10—N3	1.361 (3)
C3—C12	1.316 (3)	C10—N5	1.377 (3)
C3—C4	1.513 (3)	C11—O3	1.250 (3)
C4—N1	1.483 (2)	C11—N5	1.418 (3)
C4—C5	1.546 (3)	C12—H12A	0.9300
C4—H4	0.9800	C12—H12B	0.9300
C5—C6	1.534 (3)	N3—H3A	0.8600
C5—H5A	0.9700	N3—H3B	0.8600
C5—H5B	0.9700	N5—H5C	0.97 (3)
C6—O2	1.445 (3)	O1—H1	0.8200
C6—H6	0.9800	O2—H2A	0.8200
C7—N2	1.323 (3)	O1W—H2W	0.819 (19)
C7—N1	1.388 (3)	O1W—H1W	0.821 (12)
C7—H7	0.9300		
O1—C1—C2	109.91 (19)	N2—C7—N1	113.4 (2)
O1—C1—H1A	109.7	N2—C7—H7	123.3
C2—C1—H1A	109.7	N1—C7—H7	123.3
O1—C1—H1B	109.7	N4—C8—C9	128.1 (2)
C2—C1—H1B	109.7	N4—C8—N1	125.74 (19)
H1A—C1—H1B	108.2	C9—C8—N1	106.11 (19)
C6—C2—C3	103.73 (19)	C8—C9—N2	110.5 (2)
C6—C2—C1	110.8 (2)	C8—C9—C11	119.4 (2)
C3—C2—C1	111.62 (18)	N2—C9—C11	130.0 (2)
C6—C2—H2	110.2	N4—C10—N3	119.1 (2)
C3—C2—H2	110.2	N4—C10—N5	123.8 (2)
C1—C2—H2	110.2	N3—C10—N5	117.10 (19)
C12—C3—C4	123.0 (2)	O3—C11—N5	120.7 (2)
C12—C3—C2	127.9 (2)	O3—C11—C9	128.3 (2)
C4—C3—C2	109.04 (18)	N5—C11—C9	110.97 (18)
N1—C4—C3	114.69 (17)	C3—C12—H12A	120.0
N1—C4—C5	115.16 (18)	C3—C12—H12B	120.0
C3—C4—C5	103.58 (17)	H12A—C12—H12B	120.0
N1—C4—H4	107.7	C7—N1—C8	105.70 (17)
C3—C4—H4	107.7	C7—N1—C4	128.21 (18)
C5—C4—H4	107.7	C8—N1—C4	125.75 (18)
C6—C5—C4	103.91 (17)	C7—N2—C9	104.25 (19)
C6—C5—H5A	111.0	C10—N3—H3A	120.0
C4—C5—H5A	111.0	C10—N3—H3B	120.0
C6—C5—H5B	111.0	H3A—N3—H3B	120.0

C4—C5—H5B	111.0	C10—N4—C8	111.93 (18)
H5A—C5—H5B	109.0	C10—N5—C11	125.68 (18)
O2—C6—C2	106.42 (19)	C10—N5—H5C	118 (2)
O2—C6—C5	111.52 (19)	C11—N5—H5C	116 (2)
C2—C6—C5	102.89 (18)	C1—O1—H1	109.5
O2—C6—H6	111.8	C6—O2—H2A	109.5
C2—C6—H6	111.8	H2W—O1W—H1W	115 (2)
C5—C6—H6	111.8		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N4^{i}	0.82	2.04	2.857 (2)	172
O2—H2*A*···O1*W*^{ii}	0.82	1.83	2.639 (3)	169
N3—H3*B*···O3^{iii}	0.86	2.24	3.039 (3)	154
N5—H5*C*···N2^{iii}	0.97 (3)	1.86 (3)	2.829 (3)	177 (3)
O1*W*—H2*W*···O1^{iv}	0.82 (2)	2.11 (1)	2.900 (3)	161 (3)
O1*W*—H1*W*···O2^{v}	0.82 (1)	2.00 (2)	2.783 (3)	159 (4)

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