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

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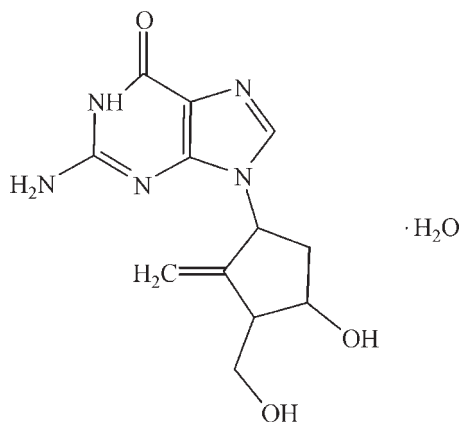
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.030; wR factor = 0.079; data-to-parameter ratio = 6.8.

In the crystal of the title compound, $\text{C}_{12}\text{H}_{15}\text{N}_5\text{O}_3 \cdot \text{H}_2\text{O}$, the component species are linked by $\text{N}-\text{H} \cdots \text{N}$, $\text{N}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{N}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For background, see: Czarnik (2008).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{15}\text{N}_5\text{O}_3 \cdot \text{H}_2\text{O}$

$M_r = 295.31$

Orthorhombic, $C222_1$
 $a = 6.9986$ (10) Å
 $b = 11.6229$ (10) Å
 $c = 33.932$ (3) Å
 $V = 2760.1$ (5) Å³

$Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 273$ K
 $0.12 \times 0.10 \times 0.08$ mm

Data collection

Bruker APEX2 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

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, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1} \cdots \text{N4}^i$	0.82	2.04	2.857 (2)	172
$\text{O2}-\text{H2A} \cdots \text{O1W}^{ii}$	0.82	1.83	2.639 (3)	169
$\text{N3}-\text{H3B} \cdots \text{O3}^{iii}$	0.86	2.24	3.039 (3)	154
$\text{N5}-\text{H5C} \cdots \text{N2}^{iii}$	0.97 (3)	1.86 (3)	2.829 (3)	177 (3)
$\text{O1W}-\text{H2W} \cdots \text{O1}^{iv}$	0.819 (19)	2.113 (10)	2.900 (3)	161 (3)
$\text{O1W}-\text{H1W} \cdots \text{O2}^v$	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*.

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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.* **A64**, 112–122.

supplementary materials

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

B. Jiang and Z. Liu

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 multi-point 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.

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 ethanol was heated to 443 K and kept at this temperature for one day. Upon cooling, colourless blocks of (I) were recovered. Anal. Calc. for C₁₂H₁₇N₅O₄: C 48.76, H 5.08, N 23.70%; Found: 48.68, H 5.05, N 23.66%.

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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (aryl, methylene) or $1.5U_{\text{eq}}(\text{C})$ (methyl). The water H atoms were refined with restraints of O—H = 0.82 (1)Å and H···H = 1.38 (1)Å.

Figures

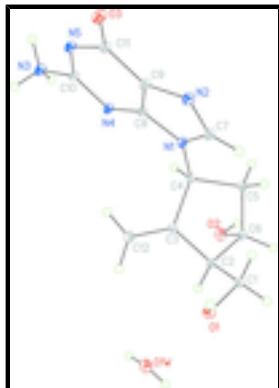


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 30% probability level.

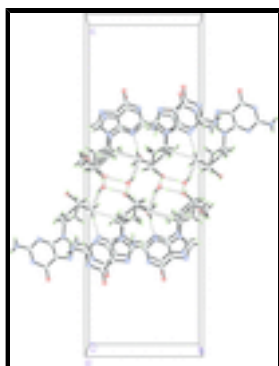


Fig. 2. A view of (I) packing structure.

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

$a = 6.9986 (10) \text{ \AA}$

$b = 11.6229 (10) \text{ \AA}$

$c = 33.932 (3) \text{ \AA}$

$V = 2760.1 (5) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1248$

$D_x = 1.421 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1377 reflections

$\theta = 3.4\text{--}25.0^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 273 \text{ K}$

Block, colorless

$0.12 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 273 \text{ K}$

1377 independent reflections

1270 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 25.0^\circ$

φ and ω scans $\theta_{\min} = 3.4^\circ$
 Absorption correction: multi-scan $h = -8 \rightarrow 8$
 (SADABS; Bruker, 2004)
 $T_{\min} = 0.987$, $T_{\max} = 0.991$ $k = -11 \rightarrow 13$
 6725 measured reflections $l = -33 \rightarrow 40$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.030$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.079$ $w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.4836P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.00$ $(\Delta/\sigma)_{\max} = 0.001$
 1377 reflections $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
 204 parameters $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$
 4 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

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	0.5675 (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*

supplementary materials

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 (\AA^2)

	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, °)

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)

supplementary materials

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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots N4 ⁱ	0.82	2.04	2.857 (2)	172
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N5—H5C \cdots N2 ⁱⁱⁱ	0.97 (3)	1.86 (3)	2.829 (3)	177 (3)
O1W—H2W \cdots O1 ^{iv}	0.819 (19)	2.113 (10)	2.900 (3)	161 (3)
O1W—H1W \cdots O2 ^v	0.821 (12)	2.000 (16)	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) $x-1/2, -y+1/2, -z+1$.

Fig. 1

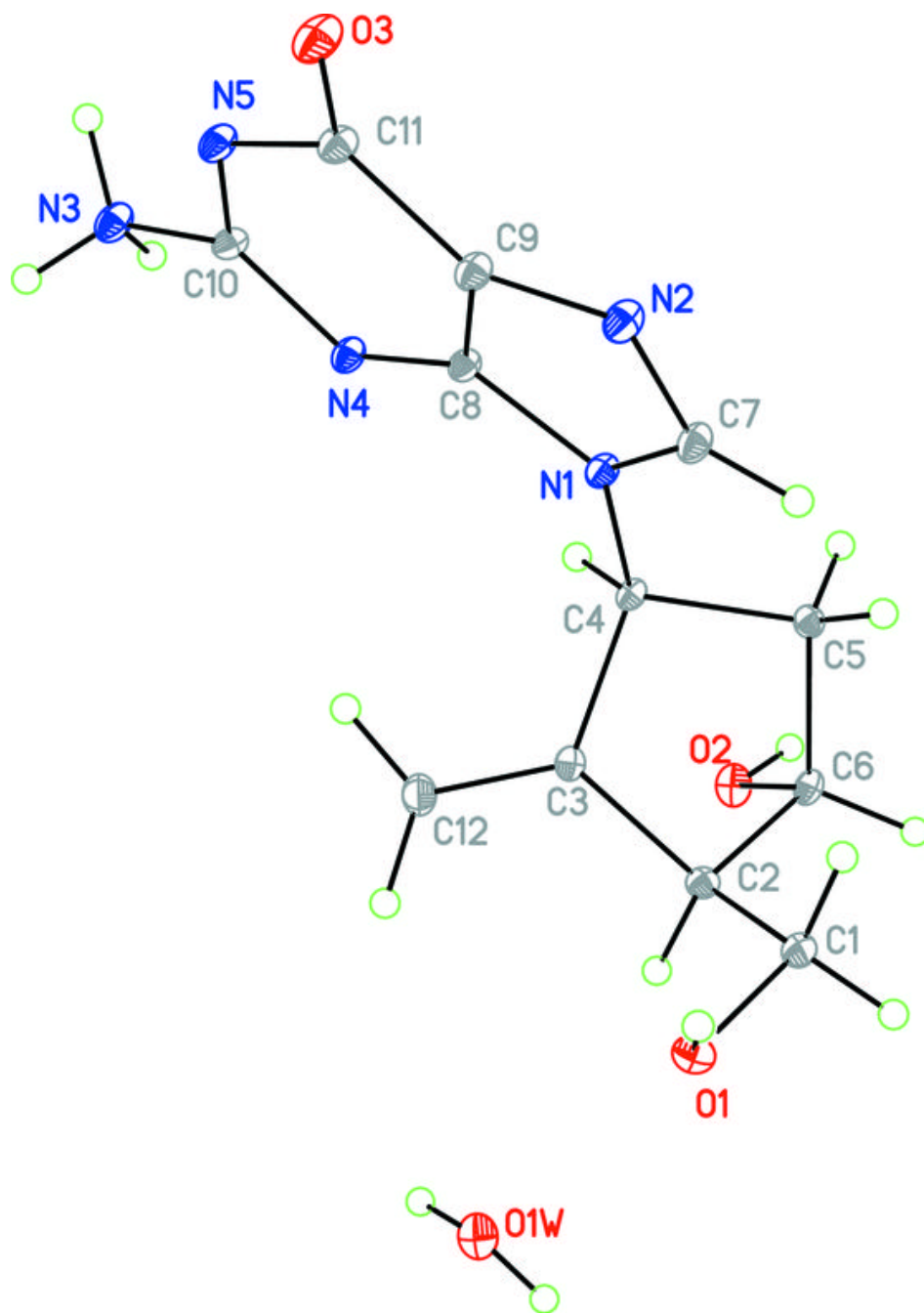


Fig. 2

