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N-(7-Dimethoxymethyl-4-oxo-3,4-dihydropteridin-2-yl)-2,2-dimethylpropionamide monohydrate

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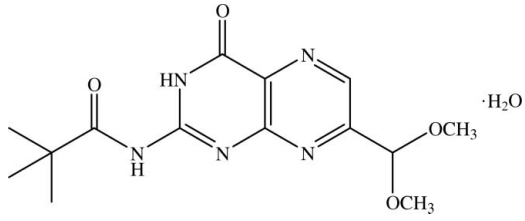
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.061; wR factor = 0.166; data-to-parameter ratio = 20.5.

In the title compound, $C_{14}H_{19}N_5O_4\cdot H_2O$, the 3,4-dihydropteridine ring system deviates sigificantly from planarity, the dihedral angle between the mean planes of the two rings being 3.93 (9)°. Intramolecular N—H···O hydrogen bonding generates an S(6) ring motif. The water molecule forms O—H···O and O—H···N intramolecular hydrogen bonds with the substituted pteridine molecule. In the crystal structure, the substituted pteridine molecules are linked by N—H···N hydrogen bonds into chains running along the c direction. These chains are further connected to the water molecules by N—H···O, O—H···O and O—H···N hydrogen bonds to form two-dimensional networks parallel to the bc plane. The crystal structure is stabilized by intra- and intermolecular N—H···O, N—H···N, O—H···O and O—H···N hydrogen bonds, together with weak C—H···O and C—H···N intra- and intermolecular interactions. C—H···π interactions are also observed.

Related literature

For related literature on the chemistry and applications of pteridine derivatives, see: *e.g.* Pateman *et al.* (1964); Piper & Montgomery (1977); Hille (1996); Taylor & Dumas (1981). For related structures, see: *e.g.* Goswami *et al.* (2000); Shanmuga Sundara Raj *et al.* (2000). For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975).



Experimental

Crystal data

$C_{14}H_{19}N_5O_4\cdot H_2O$
M_r = 339.36
Monoclinic, $P2_1/c$
a = 11.9683 (5) Å
b = 15.7252 (6) Å
c = 8.9289 (3) Å
β = 94.998 (2)°
V = 1674.06 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100.0 (1) K
0.57 × 0.09 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.943, T_{max} = 0.992
21832 measured reflections
4876 independent reflections
2723 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.166
S = 1.05
4876 reflections
238 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the C1/C2/C5/N3/C6/N2 and N5/C3/C4/N4/C5/C2 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···O2	0.83 (3)	2.48 (2)	2.995 (2)	121 (2)
O1W—H1W···N5	0.83 (3)	2.25 (3)	3.044 (2)	161 (2)
O1W—H2W···O2^{i}	0.84 (3)	2.04 (3)	2.868 (2)	170 (2)
N1—H1N1···O1W^{ii}	0.89 (2)	1.96 (2)	2.826 (2)	164.0 (18)
N2—H1N2···O1	0.838 (19)	2.06 (2)	2.667 (2)	128.8 (18)
N2—H1N2···N3^{iii}	0.838 (19)	2.62 (2)	3.008 (2)	109.9 (17)
C3—H3A···O3	0.93	2.28	2.649 (2)	103
C11—H11C···O1W^{ii}	0.96	2.59	3.471 (2)	152
C14—H14C···N4	0.96	2.62	3.176 (3)	117
C11—H11A···$Cg1^{iv}$	0.96	3.05	3.766 (2)	132
C14—H14A···$Cg2^{v}$	0.96	3.33	3.718 (2)	106

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2066).

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supplementary materials

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N-(7-Dimethoxymethyl-4-oxo-3,4-dihydropteridin-2-yl)-2,2-dimethylpropionamide monohydrate

H.-K. Fun, S. Goswami, A. C. Maity, S. Maity and S. Chantrapromma

Comment

6-Formylpterin is a useful precursor to the nutrient cofactor folic acid, anticancer drug methotrexate (Piper & Montgomery, 1977) and related pteridines like biopterin, neopterin, monapterin, umanopterin as well as precursor *Z* of molybdenum cofactor or Moco (Pateman *et al.*, 1964; Hille, 1996). These naturally occurring pteridine derivatives are mostly found in human urine and plasma, toad skins, germinating potatoes and various micro-organisms. All compounds of these heterocyclic ring systems found in nature so far, are derivatives of pterin and lumazine carrying different substituents in the 6- and/or 7- positions. One interesting aspect of the pterin system besides its biological importance is that pterins are fluorescent and thus fluorescence assay makes them detectable in biological systems even in trace amounts. We report here the hydrogen-bonding network in the crystal structure of the title compound, as an example of a 7-substituted soluble pterin derivative. We have previously reported the crystal structures of pivaloyl halopterins (Goswami *et al.*, 2000; Shanmuga Sundara Raj *et al.*, 2000), as part of our research program on solubilizing pterins and establish their X-ray structures to investigate their supramolecular network. The first general and unequivocal multistep 7-formylpterin dimethyl acetal was elegantly synthesized by Taylor (Taylor & Dumas, 1981).

In the molecules of the title compound (Fig. 1), the 3,4-dihydropteridine ring (N2–N5/C1–C6) deviates significantly from planarity with the largest deviations found for atoms N2 and N5 [0.0824 (17) and −0.0472 (16) Å, respectively], and the total puckering parameter Q = 0.144 (2)Å (Cremer & Pople, 1975). The dihedral angle between the mean planes of the two rings in the molecular structure is 3.93 (9)°. The 2,2-dimethylpropionamide substituent (O1/N1/C7–C11) is attached at atom C6, the dihedral angle between the mean plane of O1/N1/C6/C7/C8 group and the attached ring is 23.80 (9)°. The orientation of the two dimethoxymethyl groups can be indicated by the torsion angles C13—O3—C12—C4 = 168.57 (16)° and C14—O4—C12—C4 = 57.4 (2)°. The intramolecular N—H···O hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995). The water molecule forms intramolecular O—H···O and O—H···N hydrogen bonds with the 3,4-dihydropteridine (Table 1). In the crystal (Fig. 2), the substituted-pteridine molecules are linked by N—H···N hydrogen bonds (N2—H1N2···N3) into chains running along the *c* direction. These chains are further connected to the water molecules by N—H···O, O—H···O and O—H···N hydrogen bonds to form two-dimemsional networks parallel to the *bc* plane. The crystal is stabilized by intra- and intermolecular N—H···O, N—H···N, O—H···O and O—H···N hydrogen bonds together with weak C—H···O and C—H···N intra- and intermolecular interactions. C—H···π interactions further stabilized the crystal (Table 1); Cg_1 and Cg_2 are the centroids of the C1/C2/C5/N3/C6/N2 and N5/C3/C4/N4/C5/C2 rings, respectively.

Experimental

7-formylpterin dimethyl acetal (500 mg, 2.11 mmol) and 4-(dimethylamino)-pyridine (50 mg) dissolved in pivalic anhydride (5 ml) were heated at reflux under nitrogen until all of the starting material went into solution (6 h). The excess pivalic anhydride and pivalic acid were removed carefully through short-path distillation under reduced pressure. The brown product was washed well with sodium carbonate followed by water and then extracted with chloroform. The organic layer was evaporated under reduced pressure. The product was purified by silica gel (100–200 mesh) column chromatography eluting

with methanol in chloroform (5%) which yielded pure yellow crystalline solid of the title compound (492 mg, 66%, m.p. 396–397 K).

Refinement

Water H and H atoms attached to N were located in a difference map and their positions and isotropic displacement factors were refined. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Figures

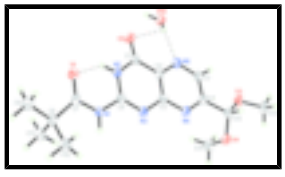

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds were dawn as dash lines.

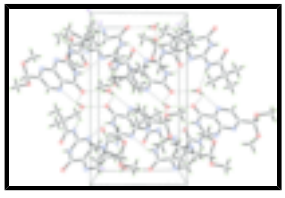

Fig. 2. The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds were drawn as dash lines.

N-(7-Dimethoxymethyl-4-oxo-3,4-dihydropteridin-2-yl)-2,2- dimethylpropionamide monohydrate

Crystal data

$C_{14}H_{19}N_5O_4 \cdot H_2O$	F_{000} = 720
M_r = 339.36	D_x = 1.346 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 396-397 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 11.9683 (5) Å	Cell parameters from 4876 reflections
b = 15.7252 (6) Å	θ = 2.6–30.0º
c = 8.9289 (3) Å	μ = 0.10 mm^{-1}
β = 94.998 (2)º	T = 100.0 (1) K
V = 1674.06 (11) Å^3	Needle, brown
Z = 4	0.57 × 0.09 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	4876 independent reflections
Radiation source: fine-focus sealed tube	2723 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.061
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 30.0º

T = 100.0(1) K	θ_{min} = 2.6º
ω scans	h = −16→14
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −17→22
T_{min} = 0.943, T_{max} = 0.992	l = −12→12
21832 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.061	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.166	$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
4876 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
238 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1W	0.33669 (13)	0.55171 (10)	0.83417 (19)	0.0319 (4)
H2W	0.3765 (19)	0.5502 (14)	0.916 (3)	0.041 (7)*
H1W	0.375 (2)	0.5259 (17)	0.776 (3)	0.064 (9)*
N1	0.74698 (13)	0.21118 (11)	0.76942 (17)	0.0226 (4)
H1N1	0.7338 (16)	0.1598 (14)	0.731 (2)	0.030 (6)*
N2	0.65491 (14)	0.33999 (10)	0.80582 (17)	0.0231 (4)
H1N2	0.6955 (18)	0.3425 (13)	0.887 (2)	0.036 (6)*
N3	0.61656 (13)	0.25861 (10)	0.58549 (16)	0.0224 (4)
N4	0.48333 (13)	0.30708 (10)	0.40139 (16)	0.0235 (4)
N5	0.42175 (13)	0.43870 (10)	0.59176 (17)	0.0280 (4)

O1	0.85318 (11)	0.29207 (9)	0.93866 (15)	0.0299 (3)
O2	0.55239 (12)	0.45420 (9)	0.86810 (15)	0.0358 (4)
O3	0.24515 (11)	0.40072 (9)	0.20062 (14)	0.0320 (4)
O4	0.32505 (11)	0.26822 (9)	0.15833 (14)	0.0301 (3)
C1	0.56955 (16)	0.39795 (12)	0.7781 (2)	0.0255 (4)
C2	0.50631 (16)	0.38486 (12)	0.6321 (2)	0.0236 (4)
C3	0.36985 (17)	0.42572 (13)	0.4568 (2)	0.0289 (5)
H3A	0.3100	0.4607	0.4241	0.035*
C4	0.40165 (16)	0.36134 (13)	0.3615 (2)	0.0251 (4)
C5	0.53529 (15)	0.31760 (12)	0.54104 (19)	0.0219 (4)
C6	0.66998 (15)	0.27179 (11)	0.7166 (2)	0.0213 (4)
C7	0.83548 (15)	0.22343 (12)	0.8770 (2)	0.0235 (4)
C8	0.90800 (16)	0.14517 (12)	0.9143 (2)	0.0249 (4)
C9	1.00897 (17)	0.17112 (14)	1.0194 (2)	0.0373 (5)
H9A	1.0516	0.2132	0.9712	0.056*
H9B	1.0551	0.1222	1.0433	0.056*
H9C	0.9839	0.1944	1.1101	0.056*
C10	0.94675 (17)	0.10571 (13)	0.7706 (2)	0.0327 (5)
H10A	0.9829	0.1484	0.7149	0.049*
H10B	0.8830	0.0836	0.7102	0.049*
H10C	0.9986	0.0604	0.7967	0.049*
C11	0.83748 (17)	0.08041 (13)	0.9936 (2)	0.0329 (5)
H11A	0.8156	0.1045	1.0854	0.049*
H11B	0.8810	0.0300	1.0158	0.049*
H11C	0.7717	0.0662	0.9291	0.049*
C12	0.34396 (16)	0.35271 (13)	0.2031 (2)	0.0273 (5)
H12A	0.3924	0.3790	0.1332	0.033*
C13	0.19087 (18)	0.41344 (14)	0.0531 (2)	0.0364 (5)
H13A	0.1329	0.4554	0.0572	0.055*
H13B	0.1585	0.3608	0.0161	0.055*
H13C	0.2448	0.4327	−0.0128	0.055*
C14	0.25935 (18)	0.22090 (15)	0.2566 (2)	0.0375 (5)
H14A	0.2477	0.1643	0.2182	0.056*
H14B	0.1882	0.2484	0.2620	0.056*
H14C	0.2983	0.2184	0.3551	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.0349 (9)	0.0347 (9)	0.0253 (8)	0.0079 (7)	−0.0017 (7)	−0.0030 (7)
N1	0.0278 (9)	0.0197 (9)	0.0194 (8)	0.0041 (7)	−0.0026 (7)	−0.0009 (7)
N2	0.0275 (9)	0.0260 (9)	0.0152 (8)	0.0036 (7)	−0.0014 (7)	−0.0015 (7)
N3	0.0236 (8)	0.0265 (9)	0.0169 (8)	0.0025 (7)	0.0012 (6)	0.0006 (6)
N4	0.0243 (9)	0.0282 (9)	0.0179 (8)	0.0028 (7)	0.0009 (7)	0.0015 (6)
N5	0.0304 (9)	0.0308 (9)	0.0223 (8)	0.0070 (7)	0.0005 (7)	−0.0010 (7)
O1	0.0286 (8)	0.0287 (8)	0.0311 (8)	0.0001 (6)	−0.0044 (6)	−0.0038 (6)
O2	0.0457 (9)	0.0352 (8)	0.0248 (7)	0.0134 (7)	−0.0058 (7)	−0.0096 (6)
O3	0.0315 (8)	0.0410 (9)	0.0227 (7)	0.0111 (6)	−0.0014 (6)	0.0005 (6)

O4	0.0330 (8)	0.0352 (9)	0.0218 (7)	0.0027 (6)	0.0016 (6)	−0.0023 (6)
C1	0.0289 (11)	0.0260 (11)	0.0214 (9)	0.0022 (8)	0.0014 (8)	0.0014 (8)
C2	0.0267 (10)	0.0230 (10)	0.0209 (9)	0.0025 (8)	0.0013 (8)	0.0019 (8)
C3	0.0328 (11)	0.0317 (12)	0.0213 (10)	0.0080 (9)	−0.0024 (8)	0.0002 (8)
C4	0.0248 (10)	0.0302 (11)	0.0203 (9)	0.0000 (8)	0.0027 (8)	0.0009 (8)
C5	0.0232 (10)	0.0244 (10)	0.0187 (9)	−0.0006 (8)	0.0041 (7)	0.0008 (8)
C6	0.0221 (10)	0.0210 (10)	0.0212 (9)	−0.0009 (8)	0.0045 (8)	0.0025 (7)
C7	0.0240 (10)	0.0278 (11)	0.0192 (9)	−0.0003 (8)	0.0039 (8)	0.0020 (8)
C8	0.0247 (10)	0.0278 (11)	0.0218 (9)	0.0024 (8)	−0.0010 (8)	0.0024 (8)
C9	0.0322 (12)	0.0365 (13)	0.0409 (13)	0.0046 (10)	−0.0098 (10)	−0.0030 (10)
C10	0.0330 (12)	0.0367 (12)	0.0281 (11)	0.0103 (9)	0.0021 (9)	−0.0002 (9)
C11	0.0339 (12)	0.0317 (12)	0.0327 (11)	0.0034 (10)	0.0011 (9)	0.0069 (9)
C12	0.0282 (11)	0.0300 (11)	0.0236 (10)	0.0031 (9)	0.0009 (8)	0.0006 (8)
C13	0.0358 (12)	0.0491 (14)	0.0229 (10)	0.0087 (11)	−0.0044 (9)	0.0077 (10)
C14	0.0374 (13)	0.0434 (14)	0.0310 (12)	−0.0070 (10)	−0.0004 (10)	0.0015 (10)

Geometric parameters (Å, °)

O1W—H2W	0.84 (2)	C3—H3A	0.9300
O1W—H1W	0.83 (3)	C4—C12	1.525 (3)
N1—C7	1.380 (2)	C7—C8	1.526 (3)
N1—C6	1.380 (2)	C8—C9	1.520 (3)
N1—H1N1	0.89 (2)	C8—C10	1.533 (3)
N2—C6	1.358 (2)	C8—C11	1.534 (3)
N2—C1	1.375 (2)	C9—H9A	0.9600
N2—H1N2	0.84 (2)	C9—H9B	0.9600
N3—C6	1.300 (2)	C9—H9C	0.9600
N3—C5	1.377 (2)	C10—H10A	0.9600
N4—C4	1.323 (2)	C10—H10B	0.9600
N4—C5	1.354 (2)	C10—H10C	0.9600
N5—C3	1.323 (2)	C11—H11A	0.9600
N5—C2	1.344 (2)	C11—H11B	0.9600
O1—C7	1.222 (2)	C11—H11C	0.9600
O2—C1	1.224 (2)	C12—H12A	0.9800
O3—C12	1.402 (2)	C13—H13A	0.9600
O3—C13	1.431 (2)	C13—H13B	0.9600
O4—C12	1.400 (2)	C13—H13C	0.9600
O4—C14	1.436 (2)	C14—H14A	0.9600
C1—C2	1.464 (3)	C14—H14B	0.9600
C2—C5	1.396 (3)	C14—H14C	0.9600
C3—C4	1.396 (3)		
H2W—O1W—H1W	104 (2)	C10—C8—C11	109.64 (17)
C7—N1—C6	126.22 (17)	C8—C9—H9A	109.5
C7—N1—H1N1	119.8 (13)	C8—C9—H9B	109.5
C6—N1—H1N1	113.9 (13)	H9A—C9—H9B	109.5
C6—N2—C1	123.54 (16)	C8—C9—H9C	109.5
C6—N2—H1N2	116.6 (15)	H9A—C9—H9C	109.5
C1—N2—H1N2	119.4 (15)	H9B—C9—H9C	109.5
C6—N3—C5	115.57 (16)	C8—C10—H10A	109.5

C4—N4—C5	116.01 (16)	C8—C10—H10B	109.5
C3—N5—C2	115.46 (16)	H10A—C10—H10B	109.5
C12—O3—C13	113.87 (15)	C8—C10—H10C	109.5
C12—O4—C14	113.63 (15)	H10A—C10—H10C	109.5
O2—C1—N2	121.69 (17)	H10B—C10—H10C	109.5
O2—C1—C2	125.64 (17)	C8—C11—H11A	109.5
N2—C1—C2	112.66 (16)	C8—C11—H11B	109.5
N5—C2—C5	122.71 (17)	H11A—C11—H11B	109.5
N5—C2—C1	117.96 (16)	C8—C11—H11C	109.5
C5—C2—C1	119.33 (17)	H11A—C11—H11C	109.5
N5—C3—C4	122.39 (18)	H11B—C11—H11C	109.5
N5—C3—H3A	118.8	O4—C12—O3	113.05 (15)
C4—C3—H3A	118.8	O4—C12—C4	113.46 (16)
N4—C4—C3	122.53 (17)	O3—C12—C4	106.26 (15)
N4—C4—C12	117.17 (16)	O4—C12—H12A	107.9
C3—C4—C12	120.28 (17)	O3—C12—H12A	107.9
N4—C5—N3	116.06 (16)	C4—C12—H12A	107.9
N4—C5—C2	120.76 (17)	O3—C13—H13A	109.5
N3—C5—C2	123.19 (16)	O3—C13—H13B	109.5
N3—C6—N2	125.05 (17)	H13A—C13—H13B	109.5
N3—C6—N1	117.33 (16)	O3—C13—H13C	109.5
N2—C6—N1	117.62 (16)	H13A—C13—H13C	109.5
O1—C7—N1	121.97 (17)	H13B—C13—H13C	109.5
O1—C7—C8	122.73 (17)	O4—C14—H14A	109.5
N1—C7—C8	115.29 (16)	O4—C14—H14B	109.5
C9—C8—C7	108.96 (16)	H14A—C14—H14B	109.5
C9—C8—C10	110.07 (17)	O4—C14—H14C	109.5
C7—C8—C10	110.55 (15)	H14A—C14—H14C	109.5
C9—C8—C11	109.47 (16)	H14B—C14—H14C	109.5
C7—C8—C11	108.11 (15)		
C6—N2—C1—O2	173.22 (18)	C5—N3—C6—N1	175.91 (15)
C6—N2—C1—C2	−7.8 (3)	C1—N2—C6—N3	9.6 (3)
C3—N5—C2—C5	−2.4 (3)	C1—N2—C6—N1	−169.62 (16)
C3—N5—C2—C1	177.85 (17)	C7—N1—C6—N3	158.04 (17)
O2—C1—C2—N5	−0.1 (3)	C7—N1—C6—N2	−22.7 (3)
N2—C1—C2—N5	−178.98 (16)	C6—N1—C7—O1	0.9 (3)
O2—C1—C2—C5	−179.84 (19)	C6—N1—C7—C8	−179.91 (16)
N2—C1—C2—C5	1.2 (3)	O1—C7—C8—C9	−7.5 (3)
C2—N5—C3—C4	−0.9 (3)	N1—C7—C8—C9	173.27 (16)
C5—N4—C4—C3	−0.2 (3)	O1—C7—C8—C10	−128.64 (19)
C5—N4—C4—C12	178.21 (16)	N1—C7—C8—C10	52.2 (2)
N5—C3—C4—N4	2.3 (3)	O1—C7—C8—C11	111.3 (2)
N5—C3—C4—C12	−176.04 (18)	N1—C7—C8—C11	−67.8 (2)
C4—N4—C5—N3	176.92 (16)	C14—O4—C12—O3	−63.6 (2)
C4—N4—C5—C2	−3.0 (3)	C14—O4—C12—C4	57.4 (2)
C6—N3—C5—N4	176.56 (16)	C13—O3—C12—O4	−66.3 (2)
C6—N3—C5—C2	−3.5 (3)	C13—O3—C12—C4	168.57 (16)
N5—C2—C5—N4	4.6 (3)	N4—C4—C12—O4	40.6 (2)
C1—C2—C5—N4	−175.67 (17)	C3—C4—C12—O4	−140.96 (18)

N5—C2—C5—N3	−175.38 (17)	N4—C4—C12—O3	165.42 (16)
C1—C2—C5—N3	4.4 (3)	C3—C4—C12—O3	−16.1 (2)
C5—N3—C6—N2	−3.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O2	0.83 (3)	2.48 (2)	2.995 (2)	121 (2)
O1W—H1W···N5	0.83 (3)	2.25 (3)	3.044 (2)	161 (2)
O1W—H2W···O2^{i}	0.84 (3)	2.04 (3)	2.868 (2)	170 (2)
N1—H1N1···O1W^{ii}	0.89 (2)	1.96 (2)	2.826 (2)	164.0 (18)
N2—H1N2···O1	0.838 (19)	2.06 (2)	2.667 (2)	128.8 (18)
N2—H1N2···N3^{iii}	0.838 (19)	2.62 (2)	3.008 (2)	109.9 (17)
C3—H3A···O3	0.93	2.28	2.649 (2)	103
C11—H11C···O1W^{ii}	0.96	2.59	3.471 (2)	152
C14—H14C···N4	0.96	2.62	3.176 (3)	117
C11—H11A···Cg1^{iv}	0.96	3.05	3.766 (2)	132
C14—H14A···Cg2^{v}	0.96	3.33	3.718 (2)	106

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

Fig. 1

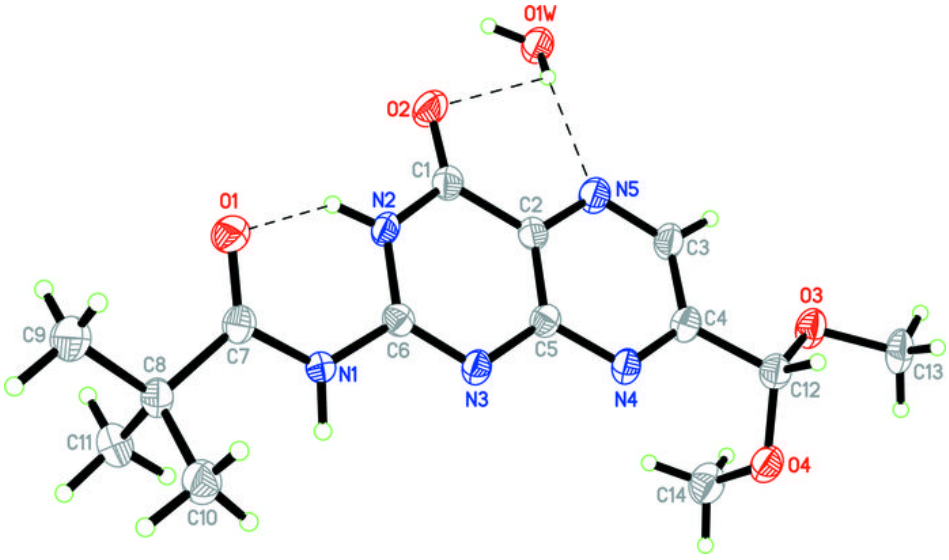
O1W
O2
C1
N5
O1
N2
C2
C3
O3
C4
C9
C8
C7
C6
C5
C13
N1
N3
N4
C12
C11
O4
C14
C10

Fig. 2