Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diammonium 1,1′,3,3′-tetramethyl-2,2′,4,4′,6,6′-hexaoxoperhydro-5,5′-bipyrimidine-5,5′-diide monohydrate

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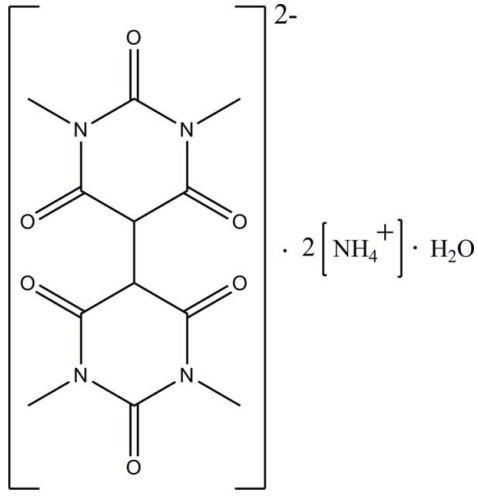
Received 30 September 2009; accepted 1 October 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.083; data-to-parameter ratio = 12.5.

In the title hydrated salt, $2NH_4^+\cdot C_{12}H_{12}N_4O_6^{2-}\cdot H_2O$, the two hexahydropyrimidine rings in the dianion are inclined to one another at a dihedral angle of 62.76 (5)°. In the crystal structure, the anions and water molecules are linked into sheets parallel to the *bc* plane by intermolecular O—H···O hydrogen bonds and sustained by C—H···O contacts. The linking of the anions and water molecules with the cations by N—H···O hydrogen bonds creates a three-dimensional extended network. The crystal structure is further stabilized by very weak C—H···π interactions.

Related literature

For general background to and applications of barbituric acid derivatives, see: Negwer (2001). For related structures, see: Rezende *et al.* (2005); da Silva *et al.* (2005). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$2NH_4^+\cdot C_{12}H_{12}N_4O_6^{2-}\cdot H_2O$
M_r = 362.36
Monoclinic, *Pc*
a = 8.5345 (1) Å
b = 12.1579 (2) Å
c = 7.7482 (1) Å
β = 100.595 (1)°
V = 790.26 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 100 K
0.46 × 0.24 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.945, T_{max} = 0.975
15091 measured reflections
3387 independent reflections
3237 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.083
S = 1.05
3387 reflections
270 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H1*N5*···O1^{i}	0.87 (2)	2.04 (2)	2.7629 (14)	141 (2)
N5—H1*N5*···O6^{i}	0.87 (2)	2.52 (2)	3.1697 (14)	132.9 (19)
N5—H2*N5*···O1^{ii}	0.85 (2)	1.97 (2)	2.8116 (14)	173 (2)
N5—H3*N5*···O5	0.94 (3)	1.90 (3)	2.8312 (14)	176.1 (19)
N5—H4*N5*···O6^{iii}	0.89 (2)	1.90 (2)	2.7819 (14)	173 (2)
N6—H1*N6*···O1*W*	0.88 (2)	2.10 (2)	2.9126 (15)	152 (2)
N6—H2*N6*···O2^{iv}	0.93 (3)	2.03 (2)	2.9074 (15)	157.8 (19)
N6—H3*N6*···O4^{v}	0.84 (2)	1.95 (2)	2.7665 (14)	164 (2)
N6—H4*N6*···O3	0.88 (2)	1.92 (2)	2.7597 (14)	158 (2)
O1*W*—H1*W*1···O3^{v}	0.84 (3)	1.96 (2)	2.7602 (13)	158 (2)
O1*W*—H2*W*1···O4^{vi}	0.78 (3)	2.01 (3)	2.7563 (14)	161 (3)
C9—H9*A*···O1*W*^{vii}	0.96	2.51	3.3658 (17)	148
C10—H10*C*···*Cg*1^{v}	0.96	2.96	3.8822 (15)	162

Symmetry codes: (i) $x-1, -y, z-\frac{1}{2}$; (ii) $x-1, y, z-1$; (iii) $x-1, y, z$; (iv) $x-1, -y+1, z-\frac{1}{2}$; (v) $x, -y+1, z-\frac{1}{2}$; (vi) $x, y, z-1$; (vii) $x+1, y, z+1$. *Cg*1 is the centroid the the C1/N1/C2/N2/C3/C4 ring.

‡ Thomson Reuters ResearcherID: A-3561-2009.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HKF and JHG thank Universiti Sains Malaysia (USM) for a Research University Golden Goose Grant (No. 1001/PFIZIK/811012). JHG thanks USM for the award of a USM Fellowship. VV is grateful to DST-India for funding through the Young Scientist Scheme (Fast Track Proposal).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2548).

References

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Negwer, M. (2001). *Organic-Chemical Drugs and their Synonyms*, 7th Rev. and Engl. Ed., Vol. 4, pp. 2873–2957. Berlin: Akademie.
Rezende, M. C., Dominguez, M., Wardell, J. L., Skakle, J. M. S., Low, J. N. & Glidewell, C. (2005). *Acta Cryst.* C**61**, o306–o311.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Silva, E. T. da, Ribiero, R. S., Lima, E. L. S., Wardell, J. L., Shakle, J. M. S., Low, J. N. & Glidewell, C. (2005). *Acta Cryst.* C**61**, o15–o20.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o2655–o2656 [https://doi.org/10.1107/S1600536809039968]

Diammonium 1,1′,3,3′-tetramethyl-2,2′,4,4′,6,6′-hexaoxoperhydro-5,5′-bipyrimidine-5,5′-diide monohydrate

Hoong-Kun Fun, Jia Hao Goh, B. Palakshi Reddy, V. Vijayakumar and S. Sarveswari

S1. Comment

Barbituric acid derivatives show high hypnotic and sedative activity (Negwer, 2001).

The asymmetric unit of the title hydrated salt (I, Fig. 1) contains two ammonium cations, a 1,1′,3,3′-tetramethyl-2,2′,4,4′,6,6′-hexaoxooctahydro-1*H*, 1′*H*-5,5′-bipyrimidine-5,5′-diide dianion and a water molecule. Two protons transfer from the C4 and C5 atoms to the ammonia molecules resulted in the formation of salts. The dianion is built up from one dimethylbarbiturate anion connected to the other one through the C_{sp^3}—C_{sp^3} (C4—C5) bond. The two hexahydropyrimidine rings are essentially planar, with maximum deviations of 0.036 (1) Å for atom C1 and 0.018 (1) Å for atom N3, respectively, for rings with atom sequence C1/N1/C2/N2/C3/C4 and C5/C6/N3/C7/N4/C8. These two rings are inclined to one another at a dihedral angle of 62.76 (5)°. The bond lengths and angles are comparable to those found in related structures (Rezende *et al.*, 2005; da Silva *et al.*, 2005).

The crystal structure of (I) is mainly stabilized by a network of N—H···O, and O—H···O hydrogen bonds as well as C—H···O and C—H···π contacts. Each ammonium H-atom participates in intermolecular hydrogen bonds. In the crystal structure (Fig. 2), the anions and water molecules are linked into sheets parallel to the *bc* plane by O—H···O hydrogen bonds and sustained by C—H···O contacts (Table 1). The ammonium cations act as bridges between the anions and water molecules *via* N—H···O hydrogen bonds (Table 1) to create a three-dimensional extended network. The crystal structure is further stabilized by weak intermolecular C10—H10C···*Cg*1 interactions (Table 1).

S2. Experimental

A solution of 1,3-dimethylbarbituric acid was refluxed in acetonitrile at 363 K for 2 h (monitored by TLC). After completion of the reaction, excess of solvent was distilled off. The solid product obtained was washed with mixture of ether and acetone, and dried. The purity of the crude product was checked through TLC and recrystallized using chloroform and benzene mixture. *M.p.* 515–517 K.

S3. Refinement

The H-atoms bound to atoms N5, N6 and O1W were located from the difference Fourier map and allowed to refine freely. The other H-atoms were placed in calculated positions, with C—H = 0.96 Å, U_{iso} = 1.5U_{eq}(C). Rotating models were used for the methyl groups. In the absence of significant anomalous dispersion, 2042 Friedel pairs were merged for the final refinement.

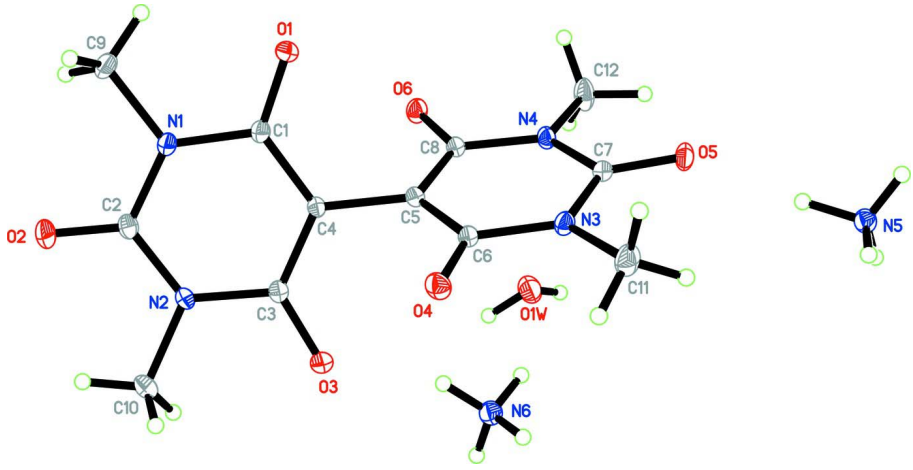


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

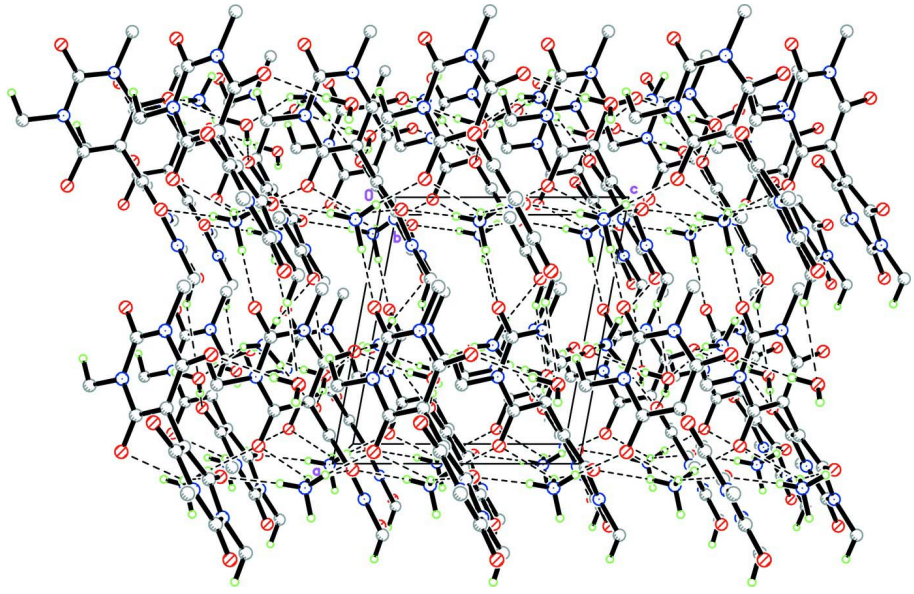


Figure 2

The crystal structure of (I) viewed along the *b* axis, showing the three-dimensional network. H atoms not involved in intermolecular interactions (dashed lines) have been omitted for clarity.

Diammonium 1,1′,3,3′-tetramethyl-2,2′,4,4′,6,6′-hexaoxoperhydro- 5,5′-bipyrimidine-5,5′-diide monohydrate

Crystal data

$2NH_4^+ \cdot C_{12}H_{12}N_4O_6^{2-} \cdot H_2O$
$M_r = 362.36$
Monoclinic, *Pc*
Hall symbol: P -2yc
$a = 8.5345$ (1) Å
$b = 12.1579$ (2) Å
$c = 7.7482$ (1) Å
$\beta = 100.595$ (1)°
$V = 790.26$ (2) Å^3
$Z = 2$
$F(000) = 384$
$D_x = 1.523$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9975 reflections
$\theta = 3.0$–34.6°
$\mu = 0.13$ mm^{-1}

T = 100 K
Block, yellow

0.46 × 0.24 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.945, T_{max} = 0.975

15091 measured reflections
3387 independent reflections
3237 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 34.7°, θ_{min} = 2.4°
h = −13→13
k = −19→19
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.083
S = 1.05
3387 reflections
270 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.0092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1)K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
O1	1.03560 (11)	0.14592 (7)	1.07661 (12)	0.01241 (15)
O2	1.29186 (11)	0.47147 (8)	1.22084 (12)	0.01554 (17)
O3	0.81723 (11)	0.48152 (7)	0.83916 (11)	0.01375 (16)
O4	0.58471 (11)	0.32057 (7)	0.97921 (12)	0.01402 (16)
O5	0.41464 (11)	0.06573 (8)	0.55640 (12)	0.01527 (17)
O6	0.94414 (10)	0.14952 (7)	0.66677 (12)	0.01358 (16)
N1	1.16599 (12)	0.30914 (8)	1.13935 (13)	0.01118 (16)
N2	1.05010 (13)	0.47550 (7)	1.03408 (14)	0.01113 (16)
N3	0.50054 (11)	0.19796 (8)	0.76033 (12)	0.01090 (16)
N4	0.67803 (12)	0.11318 (8)	0.60677 (13)	0.01124 (16)
C1	1.03474 (13)	0.24831 (9)	1.05040 (14)	0.00980 (17)

C2	1.17569 (14)	0.42182 (9)	1.13520 (15)	0.01120 (18)
C3	0.92023 (13)	0.42115 (9)	0.93130 (15)	0.01017 (17)
C4	0.91423 (13)	0.30599 (9)	0.93888 (15)	0.00967 (17)
C5	0.77985 (13)	0.24525 (9)	0.83177 (14)	0.00979 (18)
C6	0.62473 (13)	0.25855 (9)	0.86409 (14)	0.01016 (17)
C7	0.52552 (13)	0.12277 (9)	0.63627 (15)	0.01075 (18)
C8	0.80944 (13)	0.17050 (9)	0.70160 (15)	0.01024 (17)
C9	1.29964 (15)	0.25137 (11)	1.24737 (16)	0.0154 (2)
H9A	1.3982	0.2837	1.2299	0.023*
H9B	1.2972	0.1752	1.2142	0.023*
H9C	1.2912	0.2573	1.3689	0.023*
C10	1.04993 (17)	0.59583 (9)	1.03645 (19)	0.0170 (2)
H10A	1.1471	0.6218	1.1073	0.025*
H10B	0.9608	0.6216	1.0848	0.025*
H10C	1.0418	0.6231	0.9189	0.025*
C11	0.34045 (15)	0.20432 (11)	0.80192 (18)	0.0174 (2)
H11A	0.2627	0.1952	0.6964	0.026*
H11B	0.3261	0.2747	0.8530	0.026*
H11C	0.3274	0.1472	0.8837	0.026*
C12	0.70481 (16)	0.04107 (12)	0.46407 (19)	0.0201 (2)
H12A	0.6055	0.0087	0.4092	0.030*
H12B	0.7785	−0.0159	0.5103	0.030*
H12C	0.7480	0.0831	0.3789	0.030*
N5	0.08583 (12)	0.04901 (9)	0.41174 (14)	0.01257 (17)
H1N5	0.061 (3)	−0.020 (2)	0.410 (3)	0.027 (6)*
H2N5	0.067 (3)	0.083 (2)	0.315 (3)	0.029 (5)*
H3N5	0.195 (3)	0.0572 (17)	0.456 (3)	0.020 (5)*
H4N5	0.035 (3)	0.0832 (18)	0.486 (3)	0.025 (5)*
N6	0.56158 (13)	0.46002 (9)	0.56578 (14)	0.01434 (18)
H1N6	0.566 (3)	0.4075 (16)	0.488 (3)	0.016 (4)*
H2N6	0.465 (3)	0.4638 (17)	0.604 (3)	0.022 (5)*
H3N6	0.568 (3)	0.5221 (19)	0.520 (3)	0.023 (5)*
H4N6	0.641 (3)	0.4479 (18)	0.654 (3)	0.023 (5)*
O1W	0.69194 (13)	0.30906 (8)	0.33669 (14)	0.01932 (18)
H1W1	0.753 (3)	0.364 (2)	0.346 (3)	0.026 (5)*
H2W1	0.667 (3)	0.297 (2)	0.236 (4)	0.033 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0146 (4)	0.0091 (3)	0.0126 (3)	0.0000 (3)	0.0001 (3)	0.0007 (3)
O2	0.0123 (4)	0.0164 (4)	0.0170 (4)	−0.0048 (3)	0.0002 (3)	−0.0031 (3)
O3	0.0144 (4)	0.0107 (3)	0.0147 (4)	0.0010 (3)	−0.0010 (3)	0.0005 (3)
O4	0.0141 (4)	0.0137 (4)	0.0147 (4)	0.0001 (3)	0.0039 (3)	−0.0042 (3)
O5	0.0098 (3)	0.0165 (4)	0.0179 (4)	−0.0020 (3)	−0.0017 (3)	−0.0043 (3)
O6	0.0096 (3)	0.0153 (4)	0.0158 (4)	0.0003 (3)	0.0024 (3)	−0.0037 (3)
N1	0.0091 (4)	0.0118 (4)	0.0117 (4)	−0.0009 (3)	−0.0005 (3)	−0.0001 (3)
N2	0.0107 (4)	0.0087 (3)	0.0133 (4)	−0.0016 (3)	0.0002 (3)	−0.0012 (3)

N3	0.0081 (4)	0.0126 (4)	0.0119 (4)	0.0000 (3)	0.0016 (3)	−0.0014 (3)
N4	0.0092 (4)	0.0112 (4)	0.0128 (4)	−0.0003 (3)	0.0009 (3)	−0.0041 (3)
C1	0.0086 (4)	0.0109 (4)	0.0098 (4)	−0.0006 (3)	0.0014 (3)	−0.0008 (3)
C2	0.0105 (4)	0.0122 (4)	0.0110 (4)	−0.0027 (4)	0.0025 (3)	−0.0010 (3)
C3	0.0098 (4)	0.0103 (4)	0.0102 (4)	−0.0009 (3)	0.0015 (3)	−0.0010 (3)
C4	0.0090 (4)	0.0090 (4)	0.0104 (4)	−0.0004 (3)	0.0001 (3)	−0.0015 (3)
C5	0.0097 (4)	0.0089 (4)	0.0102 (4)	−0.0004 (3)	0.0002 (3)	−0.0011 (3)
C6	0.0100 (4)	0.0092 (4)	0.0107 (4)	−0.0006 (3)	0.0004 (3)	0.0001 (3)
C7	0.0096 (4)	0.0095 (4)	0.0124 (4)	−0.0003 (3)	0.0001 (3)	−0.0002 (3)
C8	0.0101 (4)	0.0090 (4)	0.0109 (4)	−0.0009 (3)	0.0001 (3)	−0.0003 (3)
C9	0.0112 (4)	0.0172 (5)	0.0160 (5)	0.0002 (4)	−0.0023 (4)	0.0023 (4)
C10	0.0188 (5)	0.0099 (4)	0.0211 (5)	−0.0024 (4)	0.0004 (4)	−0.0014 (4)
C11	0.0102 (5)	0.0222 (6)	0.0204 (5)	−0.0007 (4)	0.0044 (4)	−0.0033 (4)
C12	0.0133 (5)	0.0243 (6)	0.0224 (6)	−0.0025 (4)	0.0027 (4)	−0.0145 (5)
N5	0.0120 (4)	0.0126 (4)	0.0130 (4)	−0.0014 (3)	0.0018 (3)	0.0006 (3)
N6	0.0141 (4)	0.0137 (4)	0.0147 (4)	−0.0007 (3)	0.0013 (3)	0.0008 (3)
O1W	0.0220 (5)	0.0170 (4)	0.0176 (4)	−0.0044 (3)	0.0001 (3)	−0.0003 (3)

Geometric parameters (Å, º)

O1—C1	1.2611 (14)	C9—H9A	0.9600
O2—C2	1.2433 (14)	C9—H9B	0.9600
O3—C3	1.2611 (14)	C9—H9C	0.9600
O4—C6	1.2620 (14)	C10—H10A	0.9600
O5—C7	1.2427 (13)	C10—H10B	0.9600
O6—C8	1.2544 (14)	C10—H10C	0.9600
N1—C2	1.3733 (15)	C11—H11A	0.9600
N1—C1	1.4117 (14)	C11—H11B	0.9600
N1—C9	1.4642 (15)	C11—H11C	0.9600
N2—C2	1.3710 (15)	C12—H12A	0.9600
N2—C3	1.4052 (15)	C12—H12B	0.9600
N2—C10	1.4631 (14)	C12—H12C	0.9600
N3—C7	1.3713 (15)	N5—H1N5	0.86 (2)
N3—C6	1.4139 (14)	N5—H2N5	0.85 (3)
N3—C11	1.4623 (15)	N5—H3N5	0.94 (2)
N4—C7	1.3672 (15)	N5—H4N5	0.89 (2)
N4—C8	1.4072 (14)	N6—H1N6	0.88 (2)
N4—C12	1.4616 (16)	N6—H2N6	0.93 (2)
C1—C4	1.4033 (15)	N6—H3N6	0.84 (2)
C3—C4	1.4028 (15)	N6—H4N6	0.88 (2)
C4—C5	1.4830 (15)	O1W—H1W1	0.84 (3)
C5—C6	1.4013 (15)	O1W—H2W1	0.79 (3)
C5—C8	1.4143 (15)		
C2—N1—C1	123.86 (10)	N1—C9—H9B	109.5
C2—N1—C9	116.61 (10)	H9A—C9—H9B	109.5
C1—N1—C9	119.51 (10)	N1—C9—H9C	109.5
C2—N2—C3	123.53 (9)	H9A—C9—H9C	109.5

C2—N2—C10	118.11 (10)	H9B—C9—H9C	109.5
C3—N2—C10	118.34 (10)	N2—C10—H10A	109.5
C7—N3—C6	123.30 (9)	N2—C10—H10B	109.5
C7—N3—C11	117.50 (10)	H10A—C10—H10B	109.5
C6—N3—C11	118.70 (9)	N2—C10—H10C	109.5
C7—N4—C8	124.21 (9)	H10A—C10—H10C	109.5
C7—N4—C12	117.63 (10)	H10B—C10—H10C	109.5
C8—N4—C12	118.14 (10)	N3—C11—H11A	109.5
O1—C1—C4	125.05 (10)	N3—C11—H11B	109.5
O1—C1—N1	117.25 (10)	H11A—C11—H11B	109.5
C4—C1—N1	117.69 (9)	N3—C11—H11C	109.5
O2—C2—N2	122.47 (10)	H11A—C11—H11C	109.5
O2—C2—N1	121.16 (11)	H11B—C11—H11C	109.5
N2—C2—N1	116.36 (10)	N4—C12—H12A	109.5
O3—C3—C4	125.29 (10)	N4—C12—H12B	109.5
O3—C3—N2	116.25 (10)	H12A—C12—H12B	109.5
C4—C3—N2	118.46 (10)	N4—C12—H12C	109.5
C3—C4—C1	119.76 (10)	H12A—C12—H12C	109.5
C3—C4—C5	120.26 (9)	H12B—C12—H12C	109.5
C1—C4—C5	119.97 (9)	H1N5—N5—H2N5	117 (2)
C6—C5—C8	120.00 (9)	H1N5—N5—H3N5	109.6 (19)
C6—C5—C4	120.09 (9)	H2N5—N5—H3N5	107 (2)
C8—C5—C4	119.86 (10)	H1N5—N5—H4N5	108 (2)
O4—C6—C5	125.63 (10)	H2N5—N5—H4N5	108 (2)
O4—C6—N3	116.18 (10)	H3N5—N5—H4N5	107 (2)
C5—C6—N3	118.19 (9)	H1N6—N6—H2N6	113.9 (19)
O5—C7—N4	122.03 (11)	H1N6—N6—H3N6	110.3 (19)
O5—C7—N3	121.25 (10)	H2N6—N6—H3N6	103 (2)
N4—C7—N3	116.71 (10)	H1N6—N6—H4N6	106.3 (19)
O6—C8—N4	117.44 (10)	H2N6—N6—H4N6	111 (2)
O6—C8—C5	125.09 (10)	H3N6—N6—H4N6	112 (2)
N4—C8—C5	117.44 (10)	H1W1—O1W—H2W1	106 (3)
N1—C9—H9A	109.5		

C2—N1—C1—O1	−174.49 (10)	C3—C4—C5—C8	117.68 (12)
C9—N1—C1—O1	3.76 (15)	C1—C4—C5—C8	−63.31 (14)
C2—N1—C1—C4	5.98 (15)	C8—C5—C6—O4	178.22 (11)
C9—N1—C1—C4	−175.76 (10)	C4—C5—C6—O4	0.76 (17)
C3—N2—C2—O2	177.67 (11)	C8—C5—C6—N3	−1.73 (15)
C10—N2—C2—O2	−3.67 (17)	C4—C5—C6—N3	−179.19 (9)
C3—N2—C2—N1	−3.18 (16)	C7—N3—C6—O4	−176.44 (10)
C10—N2—C2—N1	175.47 (10)	C11—N3—C6—O4	−4.80 (15)
C1—N1—C2—O2	177.56 (10)	C7—N3—C6—C5	3.52 (15)
C9—N1—C2—O2	−0.74 (16)	C11—N3—C6—C5	175.16 (10)
C1—N1—C2—N2	−1.60 (16)	C8—N4—C7—O5	−175.66 (11)
C9—N1—C2—N2	−179.90 (10)	C12—N4—C7—O5	6.19 (17)
C2—N2—C3—O3	−176.92 (10)	C8—N4—C7—N3	3.66 (16)
C10—N2—C3—O3	4.43 (16)	C12—N4—C7—N3	−174.49 (11)

C2—N2—C3—C4	3.31 (17)	C6—N3—C7—O5	174.96 (10)
C10—N2—C3—C4	−175.34 (10)	C11—N3—C7—O5	3.22 (16)
O3—C3—C4—C1	−178.39 (11)	C6—N3—C7—N4	−4.37 (15)
N2—C3—C4—C1	1.36 (16)	C11—N3—C7—N4	−176.11 (10)
O3—C3—C4—C5	0.63 (18)	C7—N4—C8—O6	176.38 (10)
N2—C3—C4—C5	−179.62 (9)	C12—N4—C8—O6	−5.48 (16)
O1—C1—C4—C3	174.85 (11)	C7—N4—C8—C5	−2.08 (16)
N1—C1—C4—C3	−5.66 (15)	C12—N4—C8—C5	176.06 (11)
O1—C1—C4—C5	−4.17 (17)	C6—C5—C8—O6	−177.28 (11)
N1—C1—C4—C5	175.32 (9)	C4—C5—C8—O6	0.18 (17)
C3—C4—C5—C6	−64.86 (14)	C6—C5—C8—N4	1.05 (15)
C1—C4—C5—C6	114.15 (12)	C4—C5—C8—N4	178.51 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H1*N*5···O1^{i}	0.87 (2)	2.04 (2)	2.7629 (14)	141 (2)
N5—H1*N*5···O6^{i}	0.87 (2)	2.52 (2)	3.1697 (14)	132.9 (19)
N5—H2*N*5···O1^{ii}	0.85 (2)	1.97 (2)	2.8116 (14)	173 (2)
N5—H3*N*5···O5	0.94 (3)	1.90 (3)	2.8312 (14)	176.1 (19)
N5—H4*N*5···O6^{iii}	0.89 (2)	1.90 (2)	2.7819 (14)	173 (2)
N6—H1*N*6···O1*W*	0.88 (2)	2.10 (2)	2.9126 (15)	152 (2)
N6—H2*N*6···O2^{iv}	0.93 (3)	2.03 (2)	2.9074 (15)	157.8 (19)
N6—H3*N*6···O4^{v}	0.84 (2)	1.95 (2)	2.7665 (14)	164 (2)
N6—H4*N*6···O3	0.88 (2)	1.92 (2)	2.7597 (14)	158 (2)
O1*W*—H1*W*1···O3^{v}	0.84 (3)	1.96 (2)	2.7602 (13)	158 (2)
O1*W*—H2*W*1···O4^{vi}	0.78 (3)	2.01 (3)	2.7563 (14)	161 (3)
C9—H9*A*···O1*W*^{vii}	0.96	2.51	3.3658 (17)	148
C10—H10*C*···*Cg*1^{v}	0.96	2.96	3.8822 (15)	162

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