

Crystal structure of 5-hydroxy-5-propylbarbituric acid

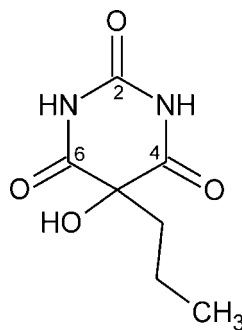
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Molecules of the title compound, $C_7H_{10}N_2O_4$, systematic name 5-hydroxy-5-propylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione, form a hydrogen-bonded framework which is based on three independent hydrogen bonds, $N-H \cdots O(\text{carbonyl})$, $N-H \cdots O(\text{hydroxy})$ and $O-H \cdots O(\text{carbonyl})$. This framework has the topology of the 5-connected **nov** net. Each molecule is linked to five other molecules *via* six hydrogen bonds, and the descriptor of the hydrogen-bonded structure is $F6_5[4^4.6^6\text{-nov}]$. The crystal packing is isostructural with that of the previously reported 5-hydroxy-5-ethyl analogue.

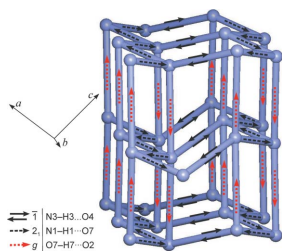
1. Chemical context

As part of a systematic investigation of solid-state properties of derivatives of barbituric acid (Gelbrich *et al.*, 2015; Zencirci *et al.*, 2014; Rossi *et al.*, 2012), we are studying the polymorphism of a group of 5-monosubstituted barbituric acids. The title compound is an oxidation product of 5-propylbarbituric acid, formed during a crystallization experiment and the structure is reported herein. The analogous oxidation product of 5-ethylbarbituric acid was previously reported by Gatehouse & Craven (1971).



2. Structural commentary

The molecule of the title compound (Fig. 1) displays a pyrimidine ring (N1/C2/N3/C4/C5/C6) in a *C5*-envelope conformation. The ring puckering parameters calculated with *PLATON* (Spek, 2009) are $\theta = 134.4$ (3), $\Phi = 52.2$ (5)° and $Q = 0.2420$ (14) Å. The distance of C5 from the mean plane defined by the other four ring atoms [maximum deviation: N3; -0.033 (1) Å] is -0.342 (2) Å. At ring atom C5 the propyl substituent adopts a *trans* conformation, and the corresponding torsion angle C5–C8–C9–C10 is -164.80 (13)°. The C5–C8–C9–C10 fragment is twisted significantly out of the plane defined by atoms C8, C5 and C2, which bisects the pyrimidinetrione fragment into two approximately symmetrical halves, resulting in a pseudo-torsion angle $C2 \cdots C5-$



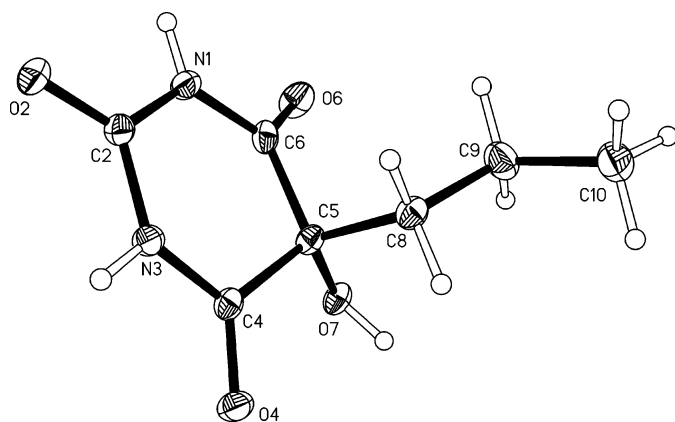


Figure 1
Asymmetric unit with displacement ellipsoids drawn at the 50% probability level and hydrogen atoms drawn as spheres of arbitrary size.

C8—C9 of $-125.69(11)^\circ$. Closer inspection suggests that this particular geometry may help to prevent unfavourably close intramolecular contacts between the O7 hydroxy group and the CH₂ group at C9, and may be also facilitate the participation of the hydroxy group in complex intermolecular hydrogen-bonding interactions.

3. Supramolecular features

One NH group and one carbonyl group of the molecule are engaged in a centrosymmetric two-point interaction, N3—H3...O4ⁱⁱ (Table 1), resulting in an $R_2^2(8)$ ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995). This kind of ring is a ubiquitous feature in crystal structures of barbiturates (Gelbrich *et al.*, 2011). The other NH group is bonded to the hydroxy group of a second molecule *via* a 2_1 operation, N1—H1...O7ⁱ, and this interaction is accompanied by a short O6...C4ⁱ contact [2.8654(18) Å]. Additionally, the hydroxy group donates a

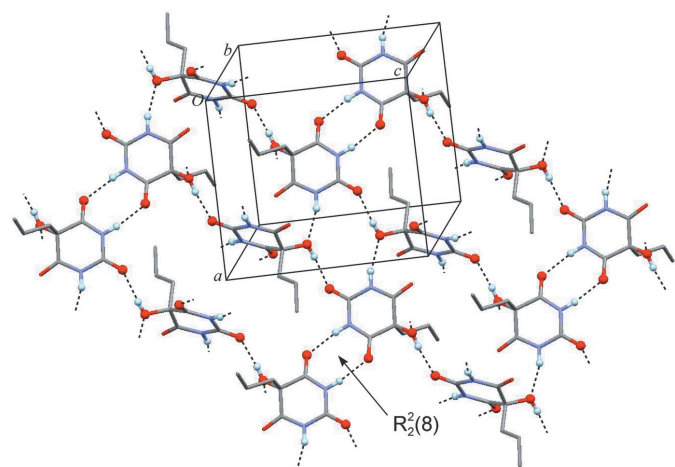


Figure 2
Layer fragment of the H-bonded framework which contains rings connecting four and six molecules in addition to $R_2^2(8)$ rings. Hydrogen bonds are drawn as dashed lines. H and O atoms engaged in hydrogen bonding are drawn as balls and all the other H atoms are omitted for clarity.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1...O7 ⁱ	0.87 (1)	2.03 (1)	2.8683 (17)	164 (2)
N3—H3...O4 ⁱⁱ	0.86 (1)	2.00 (1)	2.8451 (16)	170 (2)
O7—H7...O2 ⁱⁱⁱ	0.84 (2)	1.98 (2)	2.8055 (15)	169 (2)

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

hydrogen bond to the C2 carbonyl group of another molecule related by glide symmetry (O7—H7...O2ⁱⁱⁱ). Altogether, six hydrogen bonds connect each molecule to five other molecules. In addition to the aforementioned $R_2^2(8)$ rings, the resulting hydrogen-bonded framework structure also displays rings composed of four and six molecules (Fig. 2). This 5-connected framework has the topology of the **nov** structure (Blatov *et al.*, 2004). Fig. 3 shows a graph of the hydrogen-bonded structure (HBS) according to the methodology proposed by Hursthouse *et al.* (2015). The short descriptor according to Hursthouse *et al.* (2015) for this HBS is $F6_5[4^4.6^6\text{-nov}]$.

4. Database survey

The Cambridge Structural Database (Version 5.36; Groom & Allen, 2014) contains the crystal structure of 5,5-dihydroxybarbituric acid (Singh, 1965; Harrowfield *et al.*, 1989; CSD refcode ALXANM01) and those of a monohydrate (Lewis & Tocher, 2004; PAGYUS), a trihydrate (Lewis & Tocher, 2004b; HBARBT01) and a 1,4-dioxane hemisolvate (Gelbrich *et al.*, 2010; NUQYII) of the same compound. Two-point

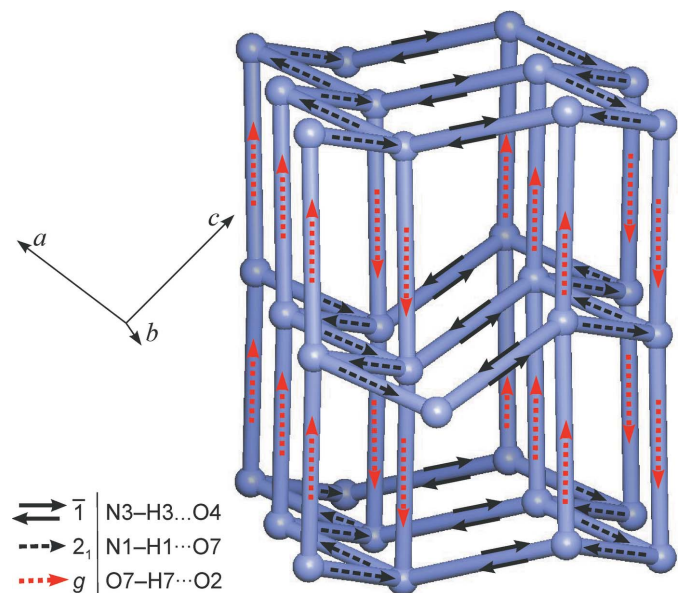


Figure 3
The N—H...O(carbonyl), N—H...O(hydroxy) and O—H...O(carbonyl) bonded $F6_5[4^4.6^6\text{-nov}]$ structure of title compound. Molecules are represented as nodes and their hydrogen-bond connections as links between them.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₇ H ₁₀ N ₂ O ₄
<i>M_r</i>	186.17
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.7862 (8), 6.7093 (5), 11.7365 (6)
β (°)	98.632 (6)
<i>V</i> (Å ³)	839.72 (10)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.12
Crystal size (mm)	0.05 × 0.05 × 0.05
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2014)
<i>T_{min}</i> , <i>T_{max}</i>	0.809, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5354, 1724, 1354
<i>R_{int}</i>	0.034
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.625
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.037, 0.095, 1.05
No. of reflections	1724
No. of parameters	138
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.29, -0.20

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2012), *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2014/6* (Sheldrick, 2015b), *XP* in *SHELXTL* (Sheldrick, 20088) and *Mercury* (Macrae *et al.*, 2006) and *pubCIF* (Westrip, 2010).

connections based on N—H···O=C bonds which result in characteristic *R*₂²(8) rings are found in each of these compounds.

The title structure displays just one such interaction which involves the carbonyl group at ring position 4 (Fig. 4). One such connection, albeit *via* the C2 carbonyl group, also exists

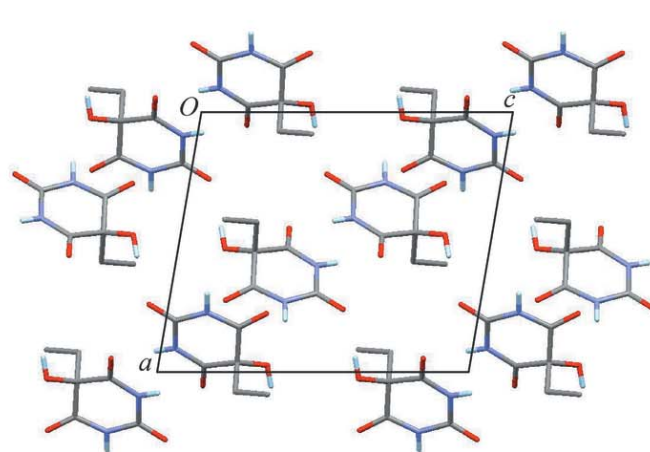
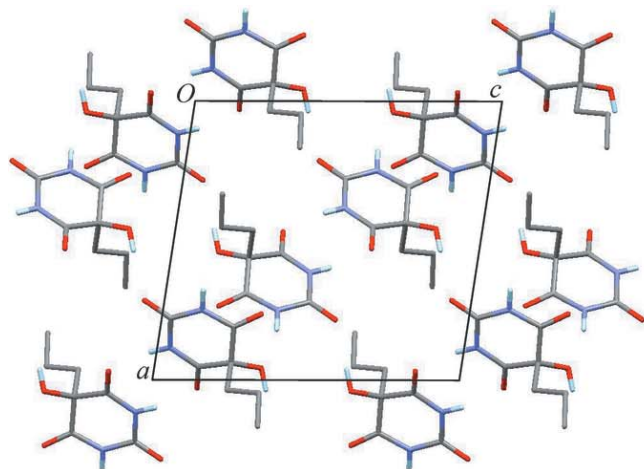


Figure 4

An illustration of the similar packing of molecules in the title compound (left) and its ethyl analogue (right). Each structure is viewed along its [010] direction. H atoms in alkyl groups are omitted for clarity.

in the 5,5-dihydroxybarbituric acid structure. Here it forms part of the C-4 ladder motif which is known from 5,5-disubstituted derivatives of barbituric acid (Gelbrich *et al.*, 2011).

The monohydrate and 1,4-dioxane hemisolvate each contain two two-point N—H···O=C connections per molecule, in the first case *via* the topologically equivalent C4 and C6 carbonyl groups and in the second *via* the C4 and C2 carbonyl groups, resulting in the looped chain motifs C-2 and C-1 (Gelbrich *et al.*, 2011), respectively, which are frequently encountered in barbiturates. C-2 chains are also found in the structure of the trihydrate. The molecular conformation of 5-hydroxy-5-ethylbarbituric acid (Gatehouse & Craven, 1971; HEBARB) is similar to that of the title structure with respect to the pseudo-torsion angle of 124.3°, which is structurally analogous to the C2···C5—C8—C9 angle discussed above. A comparison with the program *XPac* (for details, see below) indicated that these two compounds are indeed isostructural. Geometrical differences between the two molecular packing arrangements are small (Fig. 4), which is reflected in a calculated *XPac* dissimilarity index of just 5.4. This close packing similarity is remarkable insofar as the substitution of a propyl with an ethyl group alters the molecular shape considerably and leads to an 11% decrease in the volume of the unit cell. The unit-cell parameters of the two isostructures correspond directly with one another. The *a* and *b* axes of the ethyl analogue (determined at room temperature) are 6.1% and 6.5% shorter than those of the title compound. Simultaneously, the *c* axis of the ethyl analogue is 1.5% longer and the β angle is enlarged by 1.0°.

5. Synthesis and crystallization

A glass slide with a sample of 5-propylbarbituric acid embedded in paraffin oil was placed on a hot bench. The sample was melted and left to crystallize. Within a few days, the original crystals had partially converted and cube-shaped single crystals of the title compound had formed.

6. Refinement

Crystal data, data collection and structure refinement details are summarised in Table 2. The data collection was carried out in the manner described by Coles & Gale (2012). All H atoms were identified in difference maps. Methyl H atoms were idealized and included as rigid groups allowed to rotate but not tip ($C-H = 0.98 \text{ \AA}$). H atoms bonded to secondary CH_2 carbon atoms were positioned geometrically ($C-H = 0.99 \text{ \AA}$). Hydrogen atoms bonded to N atoms were refined with restrained distances [$N-H = 0.86 (1) \text{ \AA}$]. The hydrogen atom of the hydroxy group was refined freely and the U_{iso} parameters of all hydrogen atoms were also refined freely.

7. Analysis of structural features

The topology of the HBS was determined and classified with the programs *ADS* and *IsoTest* of the *TOPOS* package (Blatov, 2006) in the manner described by Baburin & Blatov (2007). The topology graph for the HBS (Fig. 3) is based on a net drawn with the *IsoCryst* program of the *TOPOS* package. The HBS of the title structure was defined by the three interactions $N-H \cdots O(\text{carbonyl})$, $N-H \cdots O(\text{hydroxy})$ and $O-H \cdots O(\text{carbonyl})$ listed in Table 1. The molecular packing in the title compound and its ethyl analogue were compared using the program *XPac* (Gelbrich & Hursthouse, 2005). The underlying calculations were based on a comparison of sets of intermolecular geometrical parameters generated from all non-H atomic positions of the title compound, except for the methyl carbon atom, and all 12 non-H atomic positions of the ethyl analogue. A match of two complete clusters consisting of a central molecule and 17 coordinating molecules was obtained with a dissimilarity index (Gelbrich *et al.*, 2012) of 5.4, indicating isostructurality of the two compounds with a high degree of packing similarity.

Acknowledgements

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supporting information

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Computing details

Data collection: *CrystalClear-SM Expert* (Rigaku, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014/6* (Sheldrick, 2015b); molecular graphics: *XP in SHELXTL* (Sheldrick, 20088) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

5-Hydroxy-5-propylpyrimidine-2,4,6(1*H*,3*H*),5*H*)-trione

Crystal data

$C_7H_{10}N_2O_4$

$M_r = 186.17$

Monoclinic, $P2_1/n$

$a = 10.7862$ (8) Å

$b = 6.7093$ (5) Å

$c = 11.7365$ (6) Å

$\beta = 98.632$ (6)°

$V = 839.72$ (10) Å³

$Z = 4$

$F(000) = 392$

$D_x = 1.473$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3013 reflections

$\theta = 2.4$ – 27.5 °

$\mu = 0.12$ mm⁻¹

$T = 100$ K

Cube, colourless

$0.05 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Saturn724+
diffractometer

Radiation source: Sealed Tube

Graphite Monochromator monochromator

Detector resolution: 28.5714 pixels mm⁻¹

profile data from ω -scans

Absorption correction: multi-scan
(*CrysAlisPro*; Agilent, 2014)

$T_{\min} = 0.809$, $T_{\max} = 1.000$

5354 measured reflections

1724 independent reflections

1354 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.4$ °

$h = -12 \rightarrow 13$

$k = -8 \rightarrow 8$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.095$

$S = 1.05$

1724 reflections

138 parameters

2 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.1441P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.29$ e Å⁻³

$\Delta\rho_{\min} = -0.20$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.75066 (12)	0.60615 (19)	0.37252 (9)	0.0154 (3)
H1	0.8189 (12)	0.536 (3)	0.3792 (16)	0.033 (5)*
O2	0.78456 (10)	0.66277 (16)	0.56534 (8)	0.0201 (3)
C2	0.72112 (13)	0.6895 (2)	0.47207 (11)	0.0147 (3)
N3	0.61584 (11)	0.80699 (19)	0.46014 (9)	0.0150 (3)
H3	0.5995 (14)	0.870 (2)	0.5200 (10)	0.020 (4)*
O4	0.47047 (9)	0.99725 (16)	0.35153 (8)	0.0180 (3)
C4	0.54700 (13)	0.8640 (2)	0.35743 (11)	0.0139 (3)
C5	0.56139 (13)	0.7360 (2)	0.25307 (11)	0.0140 (3)
O6	0.73523 (10)	0.58567 (18)	0.17883 (8)	0.0220 (3)
C6	0.68995 (13)	0.6406 (2)	0.26184 (11)	0.0153 (3)
O7	0.54144 (10)	0.85117 (16)	0.15172 (8)	0.0161 (3)
H7	0.465 (2)	0.864 (3)	0.1270 (16)	0.040 (6)*
C8	0.46287 (14)	0.5674 (2)	0.25253 (11)	0.0158 (3)
H8A	0.4849	0.4858	0.3229	0.021 (4)*
H8B	0.3799	0.6284	0.2557	0.016 (4)*
C9	0.45248 (15)	0.4312 (2)	0.14778 (13)	0.0225 (4)
H9A	0.4526	0.5128	0.0775	0.035 (5)*
H9B	0.5262	0.3417	0.1553	0.033 (5)*
C10	0.33380 (15)	0.3070 (3)	0.13545 (13)	0.0247 (4)
H10A	0.3383	0.2139	0.2003	0.034 (5)*
H10B	0.3253	0.2319	0.0631	0.036 (5)*
H10C	0.2611	0.3948	0.1350	0.044 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0131 (7)	0.0172 (7)	0.0154 (6)	0.0033 (6)	0.0002 (5)	-0.0013 (5)
O2	0.0199 (6)	0.0225 (6)	0.0159 (5)	0.0037 (5)	-0.0043 (4)	-0.0010 (4)
C2	0.0132 (8)	0.0136 (8)	0.0168 (7)	-0.0009 (6)	0.0004 (5)	0.0002 (5)
N3	0.0160 (7)	0.0173 (7)	0.0114 (6)	0.0039 (5)	0.0011 (5)	-0.0022 (5)
O4	0.0170 (6)	0.0205 (6)	0.0161 (5)	0.0058 (5)	0.0005 (4)	-0.0001 (4)
C4	0.0110 (8)	0.0155 (8)	0.0151 (7)	-0.0018 (6)	0.0016 (5)	0.0005 (5)
C5	0.0127 (8)	0.0169 (8)	0.0117 (6)	0.0010 (6)	-0.0003 (5)	0.0009 (5)
O6	0.0173 (6)	0.0323 (7)	0.0168 (5)	0.0046 (5)	0.0042 (4)	-0.0025 (5)
C6	0.0143 (8)	0.0161 (8)	0.0152 (7)	-0.0040 (6)	0.0009 (5)	-0.0003 (5)
O7	0.0143 (6)	0.0202 (6)	0.0128 (5)	-0.0006 (5)	-0.0009 (4)	0.0031 (4)
C8	0.0134 (8)	0.0178 (8)	0.0161 (6)	-0.0004 (6)	0.0021 (5)	0.0014 (6)
C9	0.0226 (9)	0.0200 (9)	0.0256 (8)	-0.0019 (7)	0.0061 (6)	-0.0065 (7)

C10	0.0262 (10)	0.0202 (9)	0.0264 (8)	-0.0030 (7)	-0.0002 (6)	0.0003 (7)
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Geometric parameters (Å, °)

N1—C2	1.3754 (18)	O6—C6	1.2110 (17)
N1—C6	1.3836 (16)	O7—H7	0.84 (2)
N1—H1	0.866 (9)	C8—C9	1.5226 (19)
O2—C2	1.2141 (16)	C8—H8A	0.9900
C2—N3	1.3720 (19)	C8—H8B	0.9900
N3—C4	1.3719 (17)	C9—C10	1.516 (2)
N3—H3	0.860 (9)	C9—H9A	0.9900
O4—C4	1.2117 (17)	C9—H9B	0.9900
C4—C5	1.5228 (19)	C10—H10A	0.9800
C5—O7	1.4076 (16)	C10—H10B	0.9800
C5—C6	1.517 (2)	C10—H10C	0.9800
C5—C8	1.551 (2)		
C2—N1—C6	126.31 (13)	N1—C6—C5	115.68 (12)
C2—N1—H1	116.4 (12)	C5—O7—H7	111.6 (14)
C6—N1—H1	116.8 (12)	C9—C8—C5	114.10 (12)
O2—C2—N3	121.52 (13)	C9—C8—H8A	108.7
O2—C2—N1	122.33 (14)	C5—C8—H8A	108.7
N3—C2—N1	116.15 (11)	C9—C8—H8B	108.7
C2—N3—C4	125.47 (12)	C5—C8—H8B	108.7
C2—N3—H3	117.8 (10)	H8A—C8—H8B	107.6
C4—N3—H3	115.6 (11)	C10—C9—C8	111.45 (13)
O4—C4—N3	122.02 (12)	C10—C9—H9A	109.3
O4—C4—C5	121.39 (11)	C8—C9—H9A	109.3
N3—C4—C5	116.26 (13)	C10—C9—H9B	109.3
O7—C5—C6	108.13 (11)	C8—C9—H9B	109.3
O7—C5—C4	110.43 (12)	H9A—C9—H9B	108.0
C6—C5—C4	112.70 (11)	C9—C10—H10A	109.5
O7—C5—C8	112.27 (11)	C9—C10—H10B	109.5
C6—C5—C8	108.15 (12)	H10A—C10—H10B	109.5
C4—C5—C8	105.19 (11)	C9—C10—H10C	109.5
O6—C6—N1	120.87 (14)	H10A—C10—H10C	109.5
O6—C6—C5	123.30 (12)	H10B—C10—H10C	109.5
C6—N1—C2—O2	-174.59 (14)	C2—N1—C6—C5	-16.4 (2)
C6—N1—C2—N3	4.9 (2)	O7—C5—C6—O6	-34.5 (2)
O2—C2—N3—C4	172.08 (14)	C4—C5—C6—O6	-156.87 (14)
N1—C2—N3—C4	-7.4 (2)	C8—C5—C6—O6	87.29 (17)
C2—N3—C4—O4	-165.40 (14)	O7—C5—C6—N1	149.92 (12)
C2—N3—C4—C5	21.1 (2)	C4—C5—C6—N1	27.56 (18)
O4—C4—C5—O7	35.46 (19)	C8—C5—C6—N1	-88.28 (15)
N3—C4—C5—O7	-151.01 (12)	O7—C5—C8—C9	54.47 (16)
O4—C4—C5—C6	156.51 (14)	C6—C5—C8—C9	-64.75 (14)
N3—C4—C5—C6	-29.96 (18)	C4—C5—C8—C9	174.60 (12)

O4—C4—C5—C8	-85.88 (16)	C5—C8—C9—C10	-164.80 (13)
N3—C4—C5—C8	87.64 (14)	C2—C5—C8—C9	-125.69 (11)
C2—N1—C6—O6	167.92 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O7 ⁱ	0.87 (1)	2.03 (1)	2.8683 (17)	164 (2)
N3—H3...O4 ⁱⁱ	0.86 (1)	2.00 (1)	2.8451 (16)	170 (2)
O7—H7...O2 ⁱⁱⁱ	0.84 (2)	1.98 (2)	2.8055 (15)	169 (2)

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