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exo,*exo*-4-(2-Hydroxyethyl)-10-oxa-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

Xue-Jie Tan,* Chen-Guang Li, Dian-Xiang Xing and Yun Liu

School of Chemical and Pharmaceutical Engineering, Shandong Institute of Light Industry, Jinan 250353, People's Republic of China
Correspondence e-mail: tanxuejie@163.com

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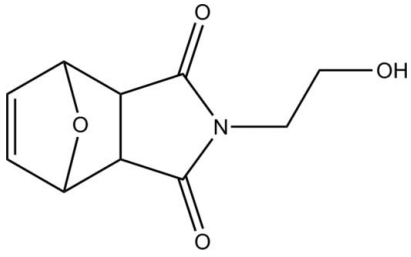
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.102; data-to-parameter ratio = 7.2.

In the crystal of the title compound, $C_{10}H_{11}NO_4$, the hydroxy group forms an O—H···$O_{carbonyl}$ hydrogen bond with an adjacent molecule, so forming chains which extend along (010). Further weak C—H···O hydrogen-bonding associations give an infinite three-dimensional network structure.

Related literature

For the first description of the title compound, see: Zhou & Chen (2000). For the synthesis of the title compound, see: Gramlich *et al.* (2010); William *et al.* (2008). For a molecular topology description, see: Braga & Grepioni (2007).



Experimental

Crystal data

$C_{10}H_{11}NO_4$
M_r = 209.20
Monoclinic, *Pc*
a = 5.4619 (12) Å
b = 6.8337 (15) Å
c = 12.546 (3) Å
β = 92.047 (3)°
V = 467.97 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.32 × 0.27 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.964, T_{max} = 0.986
2628 measured reflections
1017 independent reflections
999 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.102
S = 1.05
1017 reflections
141 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H11···O3^{i}	0.92 (6)	1.99 (6)	2.902 (3)	171 (5)
C2—H2···O3^{ii}	0.98	2.40	3.338 (3)	160
C1—H1···O4^{iii}	0.98	2.44	3.216 (3)	136

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 2000) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2178).

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supplementary materials

Acta Cryst. (2012). E**68**, o866 [doi:10.1107/S160053681200788X]

exo,*exo*-4-(2-Hydroxyethyl)-10-oxa-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

Xue-Jie Tan, Chen-Guang Li, Dian-Xiang Xing and Yun Liu

Comment

The title compound $C_{10}H_{11}NO_4$ (I), common name *N*-2-hydroxyethyl unsaturated norcantharidin (HEUNC), is a derivative of unsaturated norcantharidin, showing weaker anti-cancer activity than norcantharidin, possibly associated with its structure. However its single-crystal structure has not been unambiguously determined since first prepared in 2000 (Zhou & Chen, 2000). In the present work, the crystal structure of (I) is reported, and its molecular packing mode is discussed on the basis of its structure. The absolute configuration of (I) has not been determined.

The asymmetric unit of the title compound contains only one molecule (Fig. 1) in which the conformation of the *N*-substituted ethanol side chain is stabilized by intramolecular C—H···$O_{carbonyl}$ interactions (Table 1) [torsion angles C7—N1—C9—C10 and N1—C9—C10—O4, -73.826 (12) and 168.812 (9)°], respectively. If the ethanol side chain is not considered, the molecule has a *Z*-type configuration with planes C1, C4, C5, C6 (plane 1), C1, O1, C4 (plane 2) and C2, C3, C8, N1, C7, C9, O2, O3 (plane 3) all essentially planar [dihedral angles 50.161 (8)° between planes 1 and 2 and 53.439 (7)° between planes 2 and 3].

The hydroxy group forms intermolecular O—H···$O_{carbonyl}$ hydrogen bonds (Table 1) giving chains which extend along (010) (Fig. 2). Each molecule (shown as *O*) gives further weak C—H···O hydrogen-bonding associations with six surrounding molecules (*A*–*F*) (Fig. 2), in which molecules *B, D, E, F* and *O* are nearly co-planar, with *A* and *C* lying on either side of the plane. An overall three-dimensional network structure is formed (Fig. 3).

At first glance, the hydrogen-bonded net found in (I) may be considered distorted primitive cubic with a long vertex symbol (4.4.4.4.4.4.4.4.4.4.4.4.*.*.*.*.) (Braga & Grepioni, 2007) but there is a difference in connectivity along the sloping plane [Miller Index (0.74 0 0.67)] with 4-membered or 6-membered primitive rings occurring from each of the 15 (6*5/2) angles, actually giving a long vertex symbol of (4.4.4.4.4.4.4.4.6.6.6.6.6.6.6.6.) (Figs. 4, 5).

Experimental

For the synthesis of the title compound, see: Gramlich *et al.* (2010); William *et al.* (2008). Elemental analysis: Calcd: C 57.41; H 5.30; N 6.70%. Found: C 57.35; H 5.23; N 6.76%.

Refinement

The hydroxy H atom was located in a difference-Fourier map and both positional and isotropic displacement parameters were refined. Other H-atoms were included in calculated positions and were allowed to ride on the parent atom with C—H = 0.93–0.98 Å and with U_{iso} = 1.2U_{eq}(C). In the absence of a suitable heavy atom in the structure, Friedel pairs (757) were merged for the final stages of refinement. The described molecule has the (C1*S*,C2*R*,C3*S*,C4*R*) relative configuration.

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 2000) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

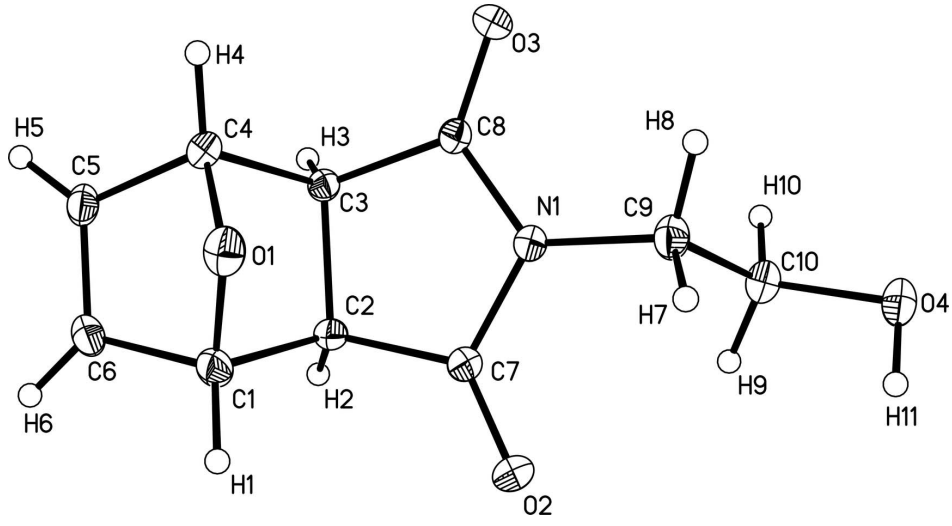

Figure 1

Molecular structure of (I) with atom numbering scheme with thermal ellipsoids drawn at the 30% probability level.

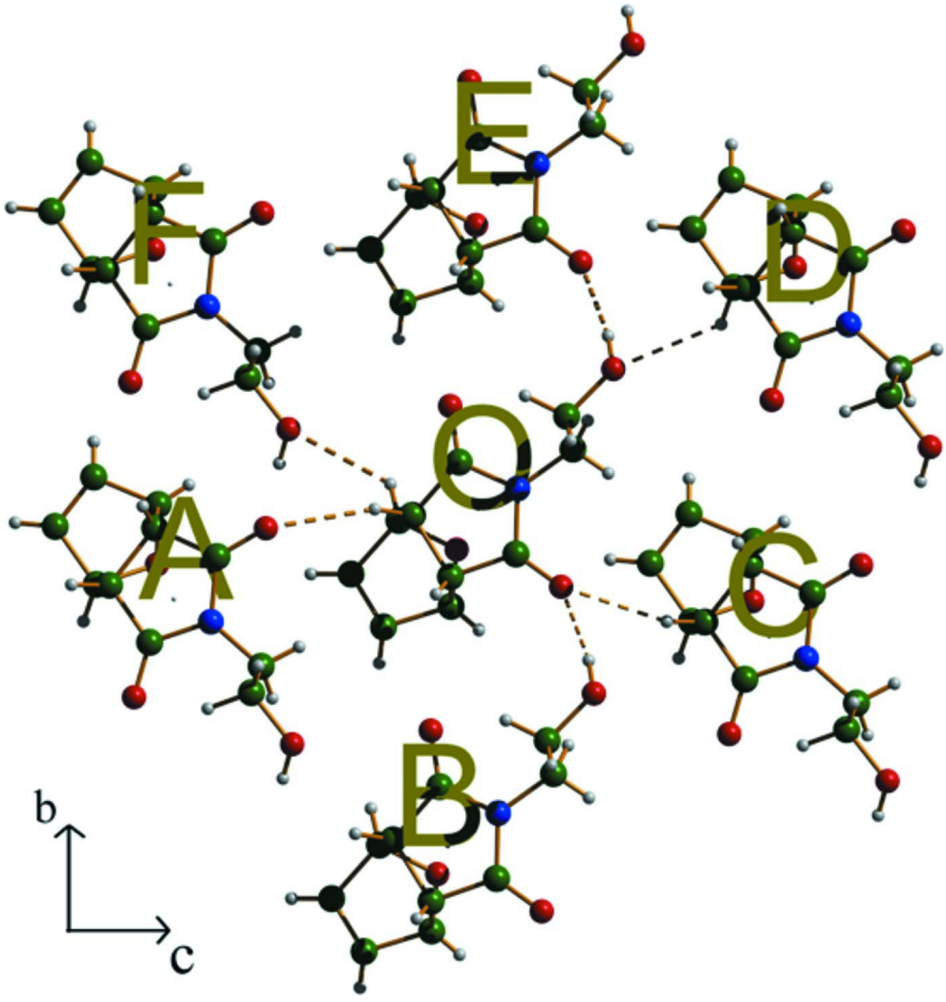

Figure 2

Hydrogen bonding (shown as dashed lines) in the crystal structure of (I) viewed along the *a* axis.

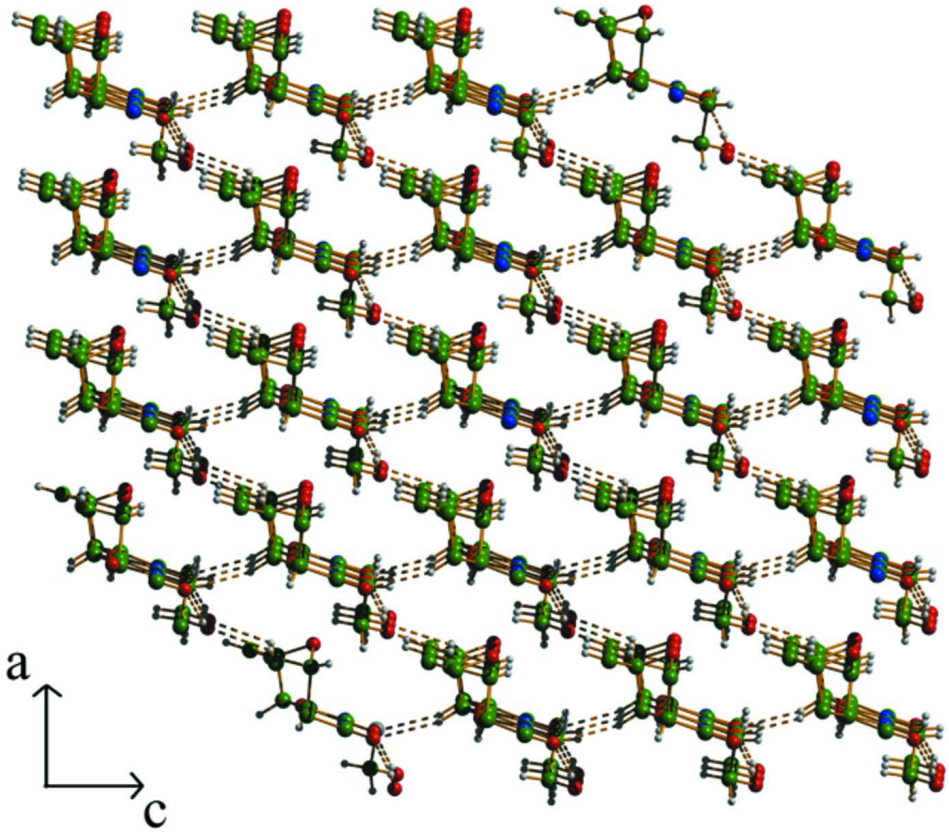

Figure 3

Portion of the infinite three-dimensional packing diagram of (I) viewed down the *b* axis.

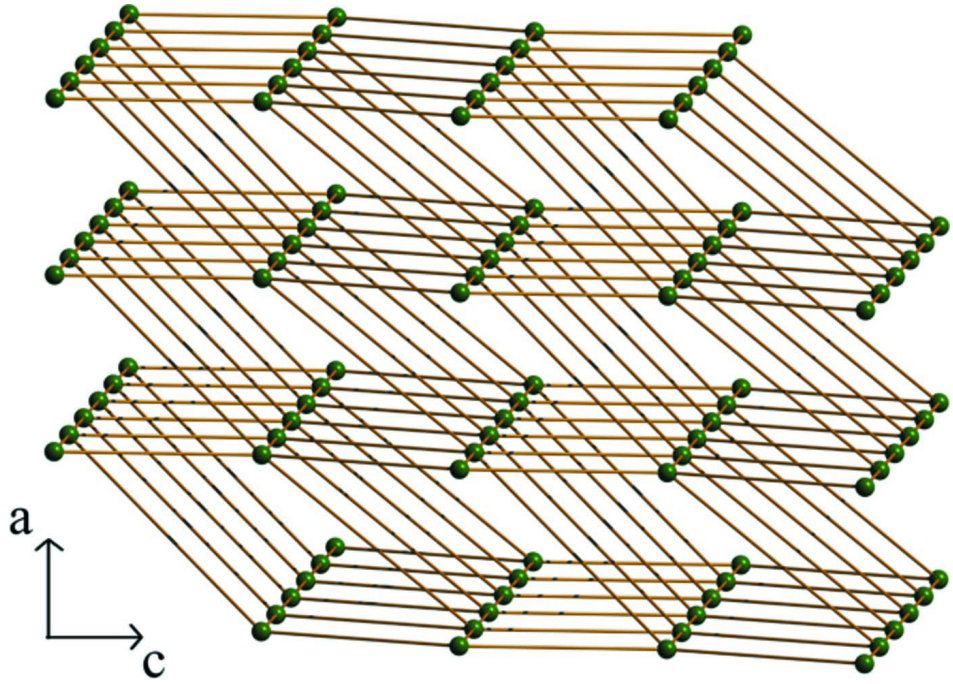

Figure 4

The (4.4.4.4.4.4.4.4.6.6.6.6.6.6.6.6.) topological diagram of (I) through abstracting every HEUNC molecule into one dummy atom viewed along the *b* axis.

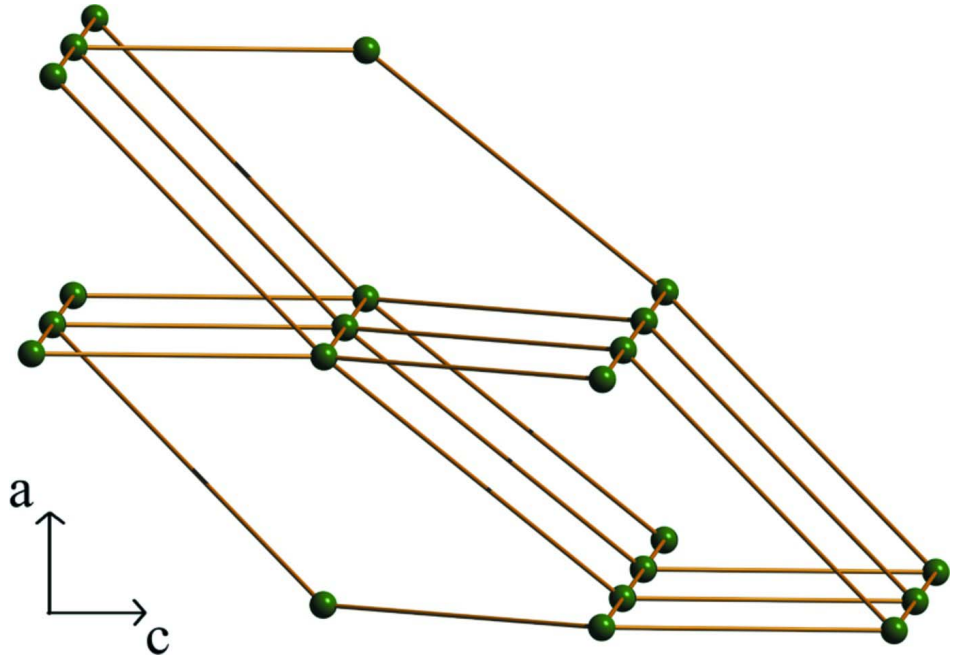

Figure 5

A sub-unit of the topology diagram from Fig. 4, showing ring generation from each angle intersection.

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Crystal data

$C_{10}H_{11}NO_4$
M_r = 209.20
Monoclinic, *Pc*
Hall symbol: P -2yc
a = 5.4619 (12) Å
b = 6.8337 (15) Å
c = 12.546 (3) Å
β = 92.047 (3)°
V = 467.97 (18) Å^3
Z = 2
F(000) = 220
D_x = 1.485 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 380 reflections
θ = 2.5–28.1°
μ = 0.12 mm^{-1}
T = 298 K
Block, colourless
0.32 × 0.27 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.964, T_{max} = 0.986
2628 measured reflections
1017 independent reflections
999 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 27.0°, θ_{min} = 3.0°
h = −6→6
k = −8→6
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.102
S = 1.05
1017 reflections
141 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
$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 0.0262P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.049 (13)

Special details

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 $F^2 > \sigma(F^2)$ 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}
C1	0.5015 (4)	0.6269 (3)	0.0567 (2)	0.0398 (5)
H1	0.5792	0.7501	0.0365	0.048*
C2	0.2181 (4)	0.6363 (3)	0.06828 (17)	0.0315 (4)

H2	0.1262	0.6354	−0.0002	0.038*
C3	0.1740 (3)	0.4531 (3)	0.13644 (16)	0.0278 (4)
H3	0.0604	0.3600	0.1017	0.033*
C4	0.4403 (4)	0.3711 (3)	0.15055 (18)	0.0343 (5)
H4	0.4673	0.2790	0.2097	0.041*
C5	0.5103 (4)	0.2973 (3)	0.0421 (2)	0.0398 (5)
H5	0.5239	0.1676	0.0207	0.048*
C6	0.5483 (5)	0.4548 (3)	−0.0159 (2)	0.0437 (6)
H6	0.5941	0.4592	−0.0866	0.052*
C7	0.1541 (4)	0.8058 (3)	0.13860 (18)	0.0343 (4)
C8	0.0818 (4)	0.5321 (3)	0.24029 (17)	0.0293 (4)
C9	0.0159 (5)	0.8636 (3)	0.3226 (2)	0.0397 (5)
H7	0.1373	0.9666	0.3310	0.048*
H8	0.0135	0.7907	0.3889	0.048*
C10	−0.2353 (5)	0.9541 (3)	0.2993 (2)	0.0431 (6)
H9	−0.2430	1.0045	0.2270	0.052*
H10	−0.3604	0.8542	0.3048	0.052*
N1	0.0834 (3)	0.7331 (2)	0.23622 (15)	0.0321 (4)
O1	0.5762 (3)	0.5517 (2)	0.15900 (16)	0.0415 (4)
O2	0.1665 (5)	0.9769 (3)	0.1182 (2)	0.0589 (6)
O3	0.0215 (3)	0.4379 (3)	0.31657 (15)	0.0429 (4)
O4	−0.2821 (4)	1.1065 (3)	0.37087 (18)	0.0548 (5)
H11	−0.181 (10)	1.203 (8)	0.347 (5)	0.099 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0380 (12)	0.0327 (12)	0.0499 (13)	−0.0054 (8)	0.0163 (9)	−0.0014 (9)
C2	0.0376 (11)	0.0285 (10)	0.0285 (9)	0.0026 (7)	0.0043 (8)	0.0035 (8)
C3	0.0282 (9)	0.0255 (10)	0.0297 (10)	−0.0006 (7)	0.0026 (7)	−0.0006 (8)
C4	0.0352 (11)	0.0303 (10)	0.0376 (11)	0.0063 (8)	0.0026 (8)	0.0019 (8)
C5	0.0375 (11)	0.0338 (11)	0.0490 (13)	0.0053 (8)	0.0118 (9)	−0.0055 (10)
C6	0.0436 (12)	0.0421 (13)	0.0466 (13)	0.0023 (10)	0.0194 (10)	−0.0026 (11)
C7	0.0361 (9)	0.0291 (10)	0.0383 (11)	0.0017 (8)	0.0075 (8)	0.0023 (9)
C8	0.0276 (8)	0.0287 (9)	0.0315 (10)	0.0022 (7)	0.0011 (6)	−0.0008 (8)
C9	0.0433 (12)	0.0393 (12)	0.0368 (10)	0.0052 (9)	0.0034 (9)	−0.0079 (9)
C10	0.0461 (13)	0.0374 (11)	0.0463 (13)	0.0080 (10)	0.0069 (10)	−0.0047 (9)
N1	0.0342 (8)	0.0286 (8)	0.0338 (9)	0.0027 (7)	0.0056 (6)	−0.0010 (7)
O1	0.0295 (7)	0.0440 (9)	0.0509 (9)	−0.0032 (7)	−0.0010 (6)	−0.0090 (7)
O2	0.0812 (14)	0.0283 (9)	0.0692 (13)	0.0041 (9)	0.0316 (11)	0.0073 (9)
O3	0.0528 (10)	0.0418 (8)	0.0348 (8)	0.0032 (8)	0.0120 (6)	0.0078 (7)
O4	0.0668 (12)	0.0420 (10)	0.0569 (12)	0.0086 (9)	0.0218 (10)	−0.0096 (9)

Geometric parameters (Å, º)

C1—O1	1.429 (3)	C5—H5	0.93
C1—C6	1.515 (3)	C6—H6	0.93
C1—C2	1.561 (3)	C7—O2	1.200 (3)
C1—H1	0.98	C7—N1	1.389 (3)
C2—C7	1.505 (3)	C8—O3	1.209 (3)

C2—C3	1.540 (3)	C8—N1	1.375 (3)
C2—H2	0.98	C9—N1	1.461 (3)
C3—C8	1.513 (3)	C9—C10	1.524 (4)
C3—C4	1.563 (3)	C9—H7	0.97
C3—H3	0.98	C9—H8	0.97
C4—O1	1.443 (3)	C10—O4	1.404 (3)
C4—C5	1.513 (3)	C10—H9	0.97
C4—H4	0.98	C10—H10	0.97
C5—C6	1.320 (4)	O4—H11	0.92 (6)
O1—C1—C6	102.24 (19)	C5—C6—C1	105.6 (2)
O1—C1—C2	100.56 (16)	C5—C6—H6	127.2
C6—C1—C2	106.08 (19)	C1—C6—H6	127.2
O1—C1—H1	115.4	O2—C7—N1	123.8 (2)
C6—C1—H1	115.4	O2—C7—C2	127.5 (2)
C2—C1—H1	115.4	N1—C7—C2	108.64 (18)
C7—C2—C3	104.83 (17)	O3—C8—N1	124.3 (2)
C7—C2—C1	109.78 (18)	O3—C8—C3	126.88 (18)
C3—C2—C1	101.17 (16)	N1—C8—C3	108.78 (17)
C7—C2—H2	113.4	N1—C9—C10	110.8 (2)
C3—C2—H2	113.4	N1—C9—H7	109.5
C1—C2—H2	113.4	C10—C9—H7	109.5
C8—C3—C2	104.57 (16)	N1—C9—H8	109.5
C8—C3—C4	111.56 (16)	C10—C9—H8	109.5
C2—C3—C4	101.02 (15)	H7—C9—H8	108.1
C8—C3—H3	112.9	O4—C10—C9	111.2 (2)
C2—C3—H3	112.9	O4—C10—H9	109.4
C4—C3—H3	112.9	C9—C10—H9	109.4
O1—C4—C5	101.84 (18)	O4—C10—H10	109.4
O1—C4—C3	100.13 (15)	C9—C10—H10	109.4
C5—C4—C3	106.37 (17)	H9—C10—H10	108.0
O1—C4—H4	115.5	C8—N1—C7	113.09 (18)
C5—C4—H4	115.5	C8—N1—C9	125.48 (19)
C3—C4—H4	115.5	C7—N1—C9	121.42 (18)
C6—C5—C4	105.9 (2)	C1—O1—C4	96.43 (16)
C6—C5—H5	127.0	C10—O4—H11	102 (3)
C4—C5—H5	127.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H11···O3^{i}	0.92 (6)	1.99 (6)	2.902 (3)	171 (5)
C2—H2···O3^{ii}	0.98	2.40	3.338 (3)	160
C1—H1···O4^{iii}	0.98	2.44	3.216 (3)	136
C9—H8···O3	0.97	2.58	2.911 (3)	100
C10—H9···O2	0.97	2.67	3.219 (3)	116

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