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3-(1-Ethyl-1*H*-pyrrole-2-carboxamido)-propionic acid monohydrate

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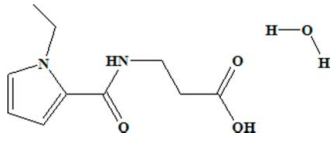
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.163; data-to-parameter ratio = 14.3.

The title compound, $C_{10}H_{14}N_2O_3\cdot H_2O$, was synthesized by alkylation of methyl 3-(1*H*-pyrrole-2-carboxamido)propionate with ethyl bromide, followed by saponification and acidification. In the crystal structure, intermolecular O—H···O and N—H···O hydrogen bonds link the molecules, forming layers parallel to the *ac* plane.

Related literature

For pyrroles sourced from marine organisms, see: Liu *et al.* (2005). For the bioactivity of pyrrole derivatives, see: Banwell *et al.* (2006); Sosa *et al.* (2002). For related structures, see: Zeng *et al.* (2005); Liu *et al.* (2006); Tang *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{14}N_2O_3\cdot H_2O$
M_r = 228.25
Monoclinic, $P2_1/c$
a = 5.2814 (12) Å
b = 31.795 (7) Å
c = 7.0226 (16) Å
β = 106.392 (4)°
V = 1131.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 K
0.47 × 0.44 × 0.15 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.953, T_{max} = 0.985
5260 measured reflections
2215 independent reflections
1772 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.163
S = 1.14
2215 reflections
155 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4^{i}	0.84	1.83	2.669 (3)	173
N2—H2···O4^{ii}	0.88	2.28	3.091 (3)	154
O4—H4*A*···O1^{iii}	0.96 (3)	1.79 (3)	2.737 (2)	170 (3)
O4—H4*B*···O2^{iv}	0.81 (3)	2.08 (4)	2.863 (3)	164 (3)

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

Data collection: *SMART* (Bruker,1999); cell refinement: *SAINT-Plus* (Bruker, 1999); 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: RZ2348).

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supporting information

Acta Cryst. (2009). E**65**, o1865 [doi:10.1107/S1600536809026749]

3-(1-Ethyl-1*H*-pyrrole-2-carboxamido)propionic acid monohydrate

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S1. Comment

Pyrrole derivatives are well known constituents of many marine organisms (Liu *et al.*, 2005), and some of them show important bioactivities, such as antitumor (Banwell *et al.*, 2006) and protein kinase inhibiting (Sosa *et al.*, 2002) activities. As a continuation of our studies in this field, which have recently resulted in the communication of the crystal structure of 3-(4-bromo-1*H*-pyrrole-2-carboxamido)propanoic acid (Zeng *et al.*, 2005), 3-(1-methyl-1H-pyrrole-2-carboxamido)propanoic acid (Liu *et al.*, 2006) and methyl 2-(1*H*-pyrrole-2-carboxamido)acetate (Tang *et al.*, 2008), we report herein the synthesis and crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), bond lengths and angles are unexceptional. In the crystal structure, molecules are linked by intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) involving water molecules to form two-dimensional layers parallel to the *ac* plane (Fig. 2, Fig. 3)

S2. Experimental

A suspension of potassium carbonate (2.10 mg, 15.0 mmol), ethyl bromide (1.87 ml, 25.0 mmol) and methyl 3-(1*H*-pyrrole-2-carbonyl)aminopropionate (0.98 g, 5.0 mmol) in acetonitrile (12 ml) was refluxed for 40 h. After evaporation of the solvent, the residue was dissolved in ethyl acetate (15 ml) and washed twice with water. The organic layer was dried over sodium sulfate and evaporated *in vacuo*. Then the alkylated product was added to a solution of 10% aqueous sodium hydroxide (10 ml) and ethanol (2 ml), and the mixture was stirred at room temperature for 24 h. The hydrolyzed mixture was made acidic with 10% hydrochloric acid to pH 2–3. After filtration, the precipitate was collected as a yellow solid (m.p. 320 K, 92.3% yield). Pale yellow crystals suitable for X-ray analysis were obtained over a period of one week by slow evaporation at room temperature of an ethanol/water solution (3:2 v/v).

S3. Refinement

All non-H atoms were refined with anisotropic displacement parameters. The water H atoms were located in a difference Fourier map and refined freely. All other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95-0.99Å, N—H = 0.88 Å, O—H = 0.84 Å, and with U_{iso} = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C, O) for methyl and hydroxy H atoms.

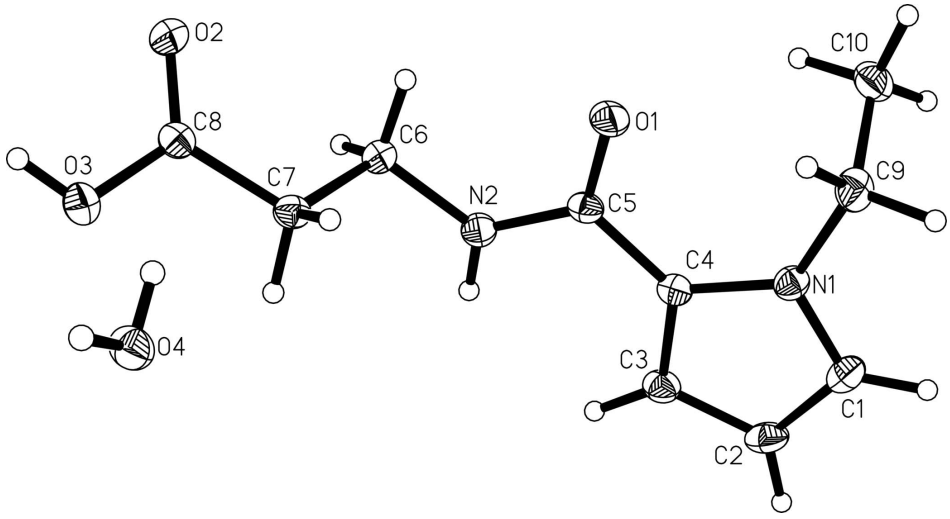

Figure 1

The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

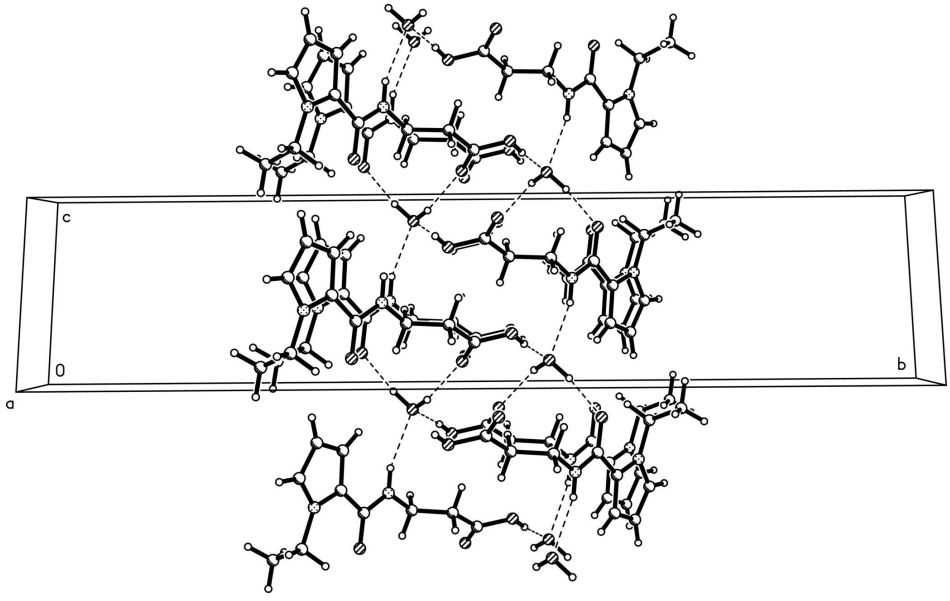

Figure 2

Crystal packing of the title compound viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

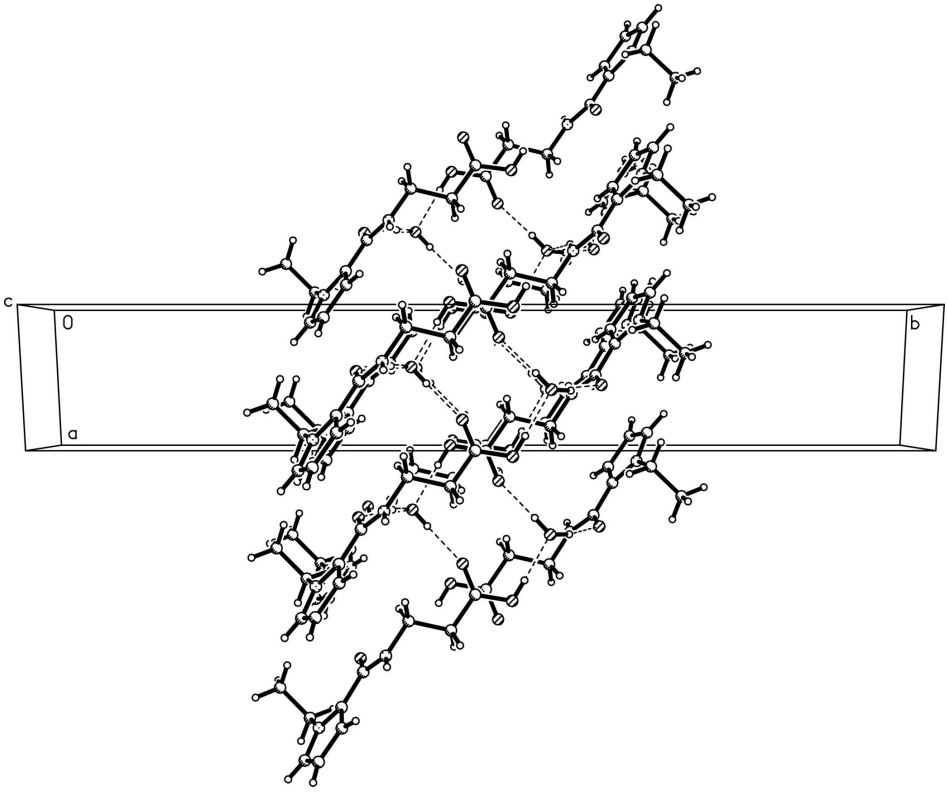

Figure 3

Crystal packing of the title compound viewed along the *c* axis. Dashed lines indicate hydrogen bonds.

3-(1-Ethyl-1*H*-pyrrole-2-carboxamido)propionic acid monohydrate

Crystal data

$C_{10}H_{14}N_2O_3 \cdot H_2O$
$M_r = 228.25$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.2814\ (12)$ Å
$b = 31.795\ (7)$ Å
$c = 7.0226\ (16)$ Å
$\beta = 106.392\ (4)°$
$V = 1131.3\ (4)$ Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.340$ Mg m^{-3}
Melting point: 320 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2595 reflections
$\theta = 2.6–28.0°$
$\mu = 0.10$ mm^{-1}
$T = 173$ K
Plate, pale yellow
0.47 × 0.44 × 0.15 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.953$, $T_{max} = 0.985$
5260 measured reflections
2215 independent reflections
1772 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.3°$
$h = -6\rightarrow4$
$k = -39\rightarrow33$
$l = -8\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.163$
$S = 1.14$
2215 reflections
155 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.087P)^2 + 0.3989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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.
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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	−0.2174 (3)	0.48016 (5)	0.1312 (3)	0.0377 (4)
O1	0.4540 (3)	0.36651 (5)	0.1693 (2)	0.0350 (4)
N1	0.9087 (4)	0.32234 (6)	0.4136 (3)	0.0281 (5)
O3	0.0271 (4)	0.53444 (5)	0.2775 (3)	0.0370 (5)
H3	−0.1105	0.5479	0.2213	0.055*
N2	0.3901 (4)	0.39704 (6)	0.4423 (3)	0.0307 (5)
H2	0.4458	0.3994	0.5723	0.037*
C4	0.7462 (4)	0.34863 (6)	0.4836 (3)	0.0254 (5)
C6	0.1614 (4)	0.42138 (7)	0.3356 (3)	0.0295 (5)
H6A	0.0205	0.4183	0.4023	0.035*
H6B	0.0936	0.4103	0.1990	0.035*
C5	0.5200 (4)	0.37122 (6)	0.3528 (3)	0.0250 (5)
C7	0.2283 (4)	0.46754 (7)	0.3265 (3)	0.0277 (5)
H7A	0.3132	0.4778	0.4627	0.033*
H7B	0.3567	0.4707	0.2485	0.033*
C8	−0.0108 (5)	0.49402 (7)	0.2343 (3)	0.0267 (5)
C3	0.8518 (5)	0.35132 (7)	0.6881 (3)	0.0319 (6)
H3A	0.7824	0.3673	0.7761	0.038*
C9	0.8797 (5)	0.30933 (8)	0.2082 (3)	0.0361 (6)
H9A	1.0516	0.2987	0.1980	0.043*
H9B	0.8319	0.3342	0.1206	0.043*
C1	1.1074 (5)	0.30880 (8)	0.5705 (4)	0.0350 (6)
H1	1.2446	0.2901	0.5629	0.042*
C10	0.6725 (6)	0.27552 (8)	0.1358 (4)	0.0407 (6)

H10A	0.7317	0.2493	0.2084	0.061*
H10B	0.6457	0.2708	−0.0065	0.061*
H10C	0.5060	0.2846	0.1585	0.061*
C2	1.0782 (5)	0.32635 (8)	0.7411 (4)	0.0366 (6)
H2A	1.1912	0.3223	0.8715	0.044*
O4	0.5768 (4)	0.57638 (6)	0.1280 (3)	0.0343 (4)
H4A	0.584 (6)	0.5980 (10)	0.034 (5)	0.057 (9)*
H4B	0.465 (7)	0.5596 (10)	0.074 (5)	0.051 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0288 (9)	0.0372 (9)	0.0415 (10)	0.0021 (7)	0.0009 (8)	−0.0031 (7)
O1	0.0450 (10)	0.0355 (9)	0.0196 (8)	0.0068 (8)	0.0013 (7)	−0.0001 (6)
N1	0.0253 (10)	0.0322 (10)	0.0270 (10)	−0.0003 (8)	0.0077 (8)	0.0016 (7)
O3	0.0413 (11)	0.0273 (8)	0.0404 (10)	0.0022 (7)	0.0087 (8)	−0.0014 (7)
N2	0.0350 (11)	0.0324 (10)	0.0220 (9)	0.0048 (8)	0.0038 (8)	0.0004 (8)
C4	0.0285 (12)	0.0239 (10)	0.0227 (11)	−0.0027 (9)	0.0057 (9)	0.0011 (8)
C6	0.0255 (12)	0.0301 (12)	0.0309 (12)	0.0011 (9)	0.0048 (9)	−0.0013 (9)
C5	0.0290 (11)	0.0225 (10)	0.0222 (11)	−0.0043 (9)	0.0049 (9)	0.0010 (8)
C7	0.0255 (11)	0.0311 (12)	0.0265 (11)	−0.0032 (9)	0.0076 (9)	−0.0026 (9)
C8	0.0311 (12)	0.0292 (11)	0.0219 (10)	−0.0009 (9)	0.0110 (9)	−0.0003 (8)
C3	0.0376 (13)	0.0312 (12)	0.0235 (11)	−0.0002 (10)	0.0029 (10)	0.0005 (9)
C9	0.0368 (14)	0.0459 (14)	0.0306 (12)	0.0049 (11)	0.0179 (11)	0.0016 (10)
C1	0.0258 (12)	0.0371 (13)	0.0393 (13)	0.0020 (10)	0.0044 (10)	0.0052 (10)
C10	0.0506 (16)	0.0392 (14)	0.0304 (13)	0.0054 (12)	0.0082 (12)	−0.0061 (10)
C2	0.0341 (13)	0.0394 (14)	0.0282 (12)	−0.0038 (11)	−0.0044 (10)	0.0039 (10)
O4	0.0431 (11)	0.0282 (9)	0.0289 (9)	−0.0011 (8)	0.0058 (8)	0.0009 (7)

Geometric parameters (Å, º)

O2—C8	1.210 (3)	C7—H7A	0.9900
O1—C5	1.245 (3)	C7—H7B	0.9900
N1—C1	1.359 (3)	C3—C2	1.395 (4)
N1—C4	1.384 (3)	C3—H3A	0.9500
N1—C9	1.466 (3)	C9—C10	1.516 (4)
O3—C8	1.323 (3)	C9—H9A	0.9900
O3—H3	0.8400	C9—H9B	0.9900
N2—C5	1.335 (3)	C1—C2	1.370 (4)
N2—C6	1.452 (3)	C1—H1	0.9500
N2—H2	0.8800	C10—H10A	0.9800
C4—C3	1.388 (3)	C10—H10B	0.9800
C4—C5	1.473 (3)	C10—H10C	0.9800
C6—C7	1.515 (3)	C2—H2A	0.9500
C6—H6A	0.9900	O4—H4A	0.96 (3)
C6—H6B	0.9900	O4—H4B	0.81 (3)
C7—C8	1.504 (3)		

C1—N1—C4	108.54 (19)	O2—C8—O3	123.0 (2)
C1—N1—C9	123.4 (2)	O2—C8—C7	124.0 (2)
C4—N1—C9	128.08 (19)	O3—C8—C7	113.00 (19)
C8—O3—H3	109.5	C4—C3—C2	107.7 (2)
C5—N2—C6	123.23 (18)	C4—C3—H3A	126.1
C5—N2—H2	118.4	C2—C3—H3A	126.1
C6—N2—H2	118.4	N1—C9—C10	113.3 (2)
N1—C4—C3	107.2 (2)	N1—C9—H9A	108.9
N1—C4—C5	123.25 (19)	C10—C9—H9A	108.9
C3—C4—C5	129.3 (2)	N1—C9—H9B	108.9
N2—C6—C7	111.60 (19)	C10—C9—H9B	108.9
N2—C6—H6A	109.3	H9A—C9—H9B	107.7
C7—C6—H6A	109.3	N1—C1—C2	109.2 (2)
N2—C6—H6B	109.3	N1—C1—H1	125.4
C7—C6—H6B	109.3	C2—C1—H1	125.4
H6A—C6—H6B	108.0	C9—C10—H10A	109.5
O1—C5—N2	122.0 (2)	C9—C10—H10B	109.5
O1—C5—C4	121.9 (2)	H10A—C10—H10B	109.5
N2—C5—C4	116.13 (18)	C9—C10—H10C	109.5
C8—C7—C6	112.51 (19)	H10A—C10—H10C	109.5
C8—C7—H7A	109.1	H10B—C10—H10C	109.5
C6—C7—H7A	109.1	C1—C2—C3	107.3 (2)
C8—C7—H7B	109.1	C1—C2—H2A	126.3
C6—C7—H7B	109.1	C3—C2—H2A	126.3
H7A—C7—H7B	107.8	H4A—O4—H4B	108 (3)
C1—N1—C4—C3	0.7 (2)	N2—C6—C7—C8	−174.39 (18)
C9—N1—C4—C3	179.4 (2)	C6—C7—C8—O2	−19.0 (3)
C1—N1—C4—C5	175.90 (19)	C6—C7—C8—O3	161.00 (19)
C9—N1—C4—C5	−5.3 (3)	N1—C4—C3—C2	−0.3 (3)
C5—N2—C6—C7	−106.2 (2)	C5—C4—C3—C2	−175.1 (2)
C6—N2—C5—O1	0.6 (3)	C1—N1—C9—C10	101.2 (3)
C6—N2—C5—C4	−179.44 (19)	C4—N1—C9—C10	−77.4 (3)
N1—C4—C5—O1	3.2 (3)	C4—N1—C1—C2	−0.8 (3)
C3—C4—C5—O1	177.3 (2)	C9—N1—C1—C2	−179.6 (2)
N1—C4—C5—N2	−176.8 (2)	N1—C1—C2—C3	0.6 (3)
C3—C4—C5—N2	−2.6 (3)	C4—C3—C2—C1	−0.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4^{i}	0.84	1.83	2.669 (3)	173
N2—H2···O4^{ii}	0.88	2.28	3.091 (3)	154
O4—H4*A*···O1^{iii}	0.96 (3)	1.79 (3)	2.737 (2)	170 (3)
O4—H4*B*···O2^{iv}	0.81 (3)	2.08 (4)	2.863 (3)	164 (3)

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