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5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

Mohamed Rida,[a] Abdusalam Alsubari,[a] El Mokhtar Essassi,[a] Hafid Zouihri[b] and Seik Weng Ng[c,d]*

[a]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, [b]Institute of Nanomaterials and Nanotechnology MAScIR, Avenue de l'Armée Royale, Rabat, Morocco, [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and [d]Chemistry Department, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia
Correspondence e-mail: seikweng@um.edu.my

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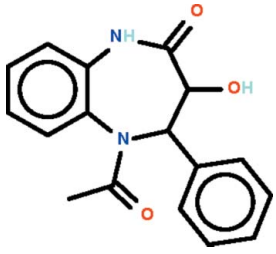
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.149; data-to-parameter ratio = 20.2.

In the title compound, $C_{17}H_{16}N_2O_3$, the seven-membered diazepine ring adopts a boat conformation with the hydroxy-substituted C atom at the prow and fused benzene ring C atoms at the stern. The phenyl substituent occupies an equatorial position. The amino group of the ring system is a hydrogen-bond donor to the oxo O atom of an inversion-related molecule, and the hydroxy group is a hydrogen-bond donor to the acetyl O atom of another inversion-related molecule. The two hydrogen bonds generate a ribbon motif parallel to [10$\bar{1}$] in the crystal structure.

Related literature

For a related 1,5-benzodiazepin-2(3*H*)-one structure, see: Rida *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{16}N_2O_3$
M_r = 296.32
Triclinic, $P\bar{1}$
a = 8.9710 (1) Å
b = 9.3142 (1) Å
c = 9.4129 (1) Å
α = 81.563 (1)°
β = 68.921 (1)°
γ = 80.146 (1)°
V = 719.95 (1) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.29 × 0.23 × 0.18 mm

Data collection

Bruker APEX DUO diffractometer
19110 measured reflections
4203 independent reflections
3584 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.149
S = 1.02
4203 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.89 (2)	2.04 (2)	2.924 (1)	175 (2)
O2—H2···O3^{ii}	0.83 (2)	2.09 (2)	2.905 (1)	168 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5376).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supporting information

Acta Cryst. (2011). E**67**, o3337 [https://doi.org/10.1107/S1600536811047878]

5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

Mohamed Rida, Abdusalam Alsubari, El Mokhtar Essassi, Hafid Zouihri and Seik Weng Ng

S1. Comment

The report on 3-hydroxy-4-phenyl-1-[(3-phenyl-4,5-dihydro-1,2-oxazol-5-yl)methyl]-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one provides the preparation and biological activity of this class of benzodiazepin-2-ones (Rida *et al.*, 2011). In the present study, the reactant, 3-ydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one, has two amino –NH– units in the ring system; however, only one site is acetylated when the compound is treated with acetic anhydride. In the title compound, the seven-membered diazepine ring adopts a boat conformation with the hydroxy-substituted C atom at the prow and fused-ring C atoms at the stern; the phenyl substituent occupies an equatorial position (Fig. 1). The amino group of the ring system is a hydrogen-bond donor to the oxo O atom of an inversion-related molecule, and the hydroxy group is hydrogen-bond donor to the acetyl O atom of another inversion-related molecule (Table 1). The two hydrogen bonds generate a ribbon motif parallel to [1 0 - 1] (Fig. 2).

S2. Experimental

3-Hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one (1 g. 3.9 mmol) was heated in acetic anhydride (20 ml) for 12 h. The precipitate was collected and recrystallized from ethanol to afford colorless crystals.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C). The amino and hydroxy H-atoms were located in a difference Fourier map and were freely refined.

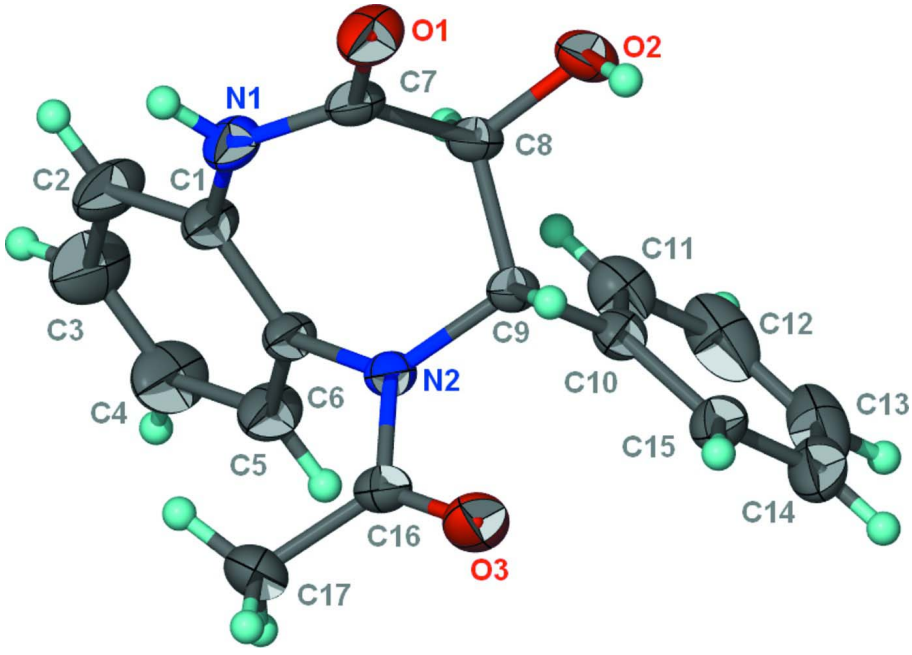

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{17}H_{16}N_2O_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

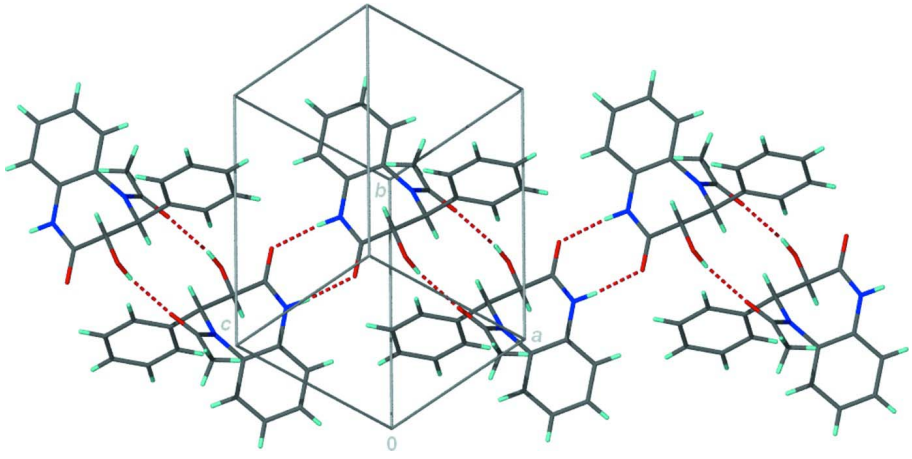

Figure 2

Ribbon motif.

5-Acetyl-3-hydroxy-4-phenyl-4,5-dihydro-1*H*-1,5-benzodiazepin- 2(3*H*)-one

Crystal data

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$c = 9.4129$ (1) Å
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$\beta = 68.921$ (1)°
$\gamma = 80.146$ (1)°
$V = 719.95$ (1) Å^3
$Z = 2$
$F(000) = 312$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9950 reflections

θ = 2.2–34.5°
μ = 0.10 mm^{-1}
T = 293 K

Prism, colorless
0.29 × 0.23 × 0.18 mm

Data collection

Bruker APEX DUO diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
19110 measured reflections
4203 independent reflections

3584 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 30.0°, θ_{min} = 2.2°
h = −12→12
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.149
S = 1.02
4203 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2 + 0.1414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.18813 (12)	0.41278 (9)	0.39332 (11)	0.0488 (2)
O2	0.50968 (12)	0.39185 (9)	0.34285 (10)	0.0430 (2)
O3	0.41433 (13)	0.74792 (11)	−0.06449 (10)	0.0513 (2)
N1	0.14350 (13)	0.64817 (10)	0.44597 (12)	0.0426 (2)
N2	0.37829 (11)	0.75893 (9)	0.18338 (9)	0.03178 (19)
C1	0.19921 (15)	0.78134 (12)	0.44620 (13)	0.0406 (3)
C2	0.1358 (2)	0.85962 (16)	0.57413 (16)	0.0656 (5)
H2A	0.0547	0.8253	0.6599	0.079*
C3	0.1927 (3)	0.98823 (18)	0.5745 (2)	0.0756 (6)
H3	0.1499	1.0396	0.6609	0.091*
C4	0.3123 (2)	1.04144 (16)	0.4483 (2)	0.0639 (4)
H4	0.3501	1.1280	0.4496	0.077*
C5	0.37548 (17)	0.96508 (13)	0.31996 (15)	0.0468 (3)
H5	0.4570	0.9999	0.2349	0.056*
C6	0.31790 (13)	0.83653 (11)	0.31723 (12)	0.0346 (2)
C7	0.24114 (14)	0.52550 (11)	0.39399 (12)	0.0363 (2)
C8	0.42230 (13)	0.53221 (11)	0.34285 (11)	0.0329 (2)
H8	0.4407	0.5843	0.4171	0.039*
C9	0.48415 (12)	0.61900 (10)	0.18549 (10)	0.0293 (2)
H9	0.4758	0.5617	0.1099	0.035*
C10	0.65889 (13)	0.64317 (11)	0.13784 (12)	0.0345 (2)
C11	0.72509 (17)	0.67606 (14)	0.24003 (18)	0.0493 (3)

H11	0.6625	0.6825	0.3425	0.059*
C12	0.8864 (2)	0.69927 (17)	0.1874 (3)	0.0685 (5)
H12	0.9311	0.7212	0.2555	0.082*
C13	0.98046 (18)	0.69010 (16)	0.0359 (3)	0.0722 (6)
H13	1.0876	0.7066	0.0020	0.087*
C14	0.91519 (18)	0.65653 (16)	−0.0649 (2)	0.0624 (4)
H14	0.9786	0.6493	−0.1671	0.075*
C15	0.75549 (15)	0.63361 (13)	−0.01454 (15)	0.0450 (3)
H15	0.7120	0.6115	−0.0835	0.054*
C16	0.35154 (14)	0.81314 (12)	0.05077 (12)	0.0371 (2)
C17	0.24042 (18)	0.95367 (15)	0.05114 (17)	0.0529 (3)
H17A	0.1983	0.9597	−0.0303	0.079*
H17B	0.2988	1.0350	0.0367	0.079*
H17C	0.1532	0.9560	0.1472	0.079*
H1	0.042 (2)	0.635 (2)	0.498 (2)	0.057 (5)*
H2	0.517 (2)	0.353 (2)	0.266 (2)	0.065 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0575 (5)	0.0342 (4)	0.0491 (5)	−0.0188 (4)	−0.0046 (4)	−0.0074 (3)
O2	0.0622 (5)	0.0290 (4)	0.0370 (4)	0.0006 (3)	−0.0196 (4)	−0.0017 (3)
O3	0.0650 (6)	0.0559 (6)	0.0302 (4)	0.0024 (4)	−0.0175 (4)	−0.0038 (4)
N1	0.0456 (5)	0.0311 (5)	0.0406 (5)	−0.0124 (4)	0.0015 (4)	−0.0043 (4)
N2	0.0399 (4)	0.0259 (4)	0.0254 (4)	−0.0040 (3)	−0.0067 (3)	−0.0015 (3)
C1	0.0511 (6)	0.0282 (5)	0.0330 (5)	−0.0082 (4)	−0.0009 (4)	−0.0050 (4)
C2	0.0924 (12)	0.0422 (7)	0.0385 (6)	−0.0147 (7)	0.0118 (7)	−0.0120 (5)
C3	0.1156 (15)	0.0465 (8)	0.0507 (8)	−0.0142 (9)	−0.0023 (9)	−0.0248 (6)
C4	0.0897 (11)	0.0368 (6)	0.0631 (9)	−0.0180 (7)	−0.0139 (8)	−0.0185 (6)
C5	0.0571 (7)	0.0316 (5)	0.0465 (6)	−0.0148 (5)	−0.0066 (5)	−0.0058 (4)
C6	0.0432 (5)	0.0258 (4)	0.0298 (5)	−0.0061 (4)	−0.0052 (4)	−0.0040 (3)
C7	0.0491 (6)	0.0288 (5)	0.0270 (4)	−0.0114 (4)	−0.0062 (4)	−0.0004 (3)
C8	0.0469 (5)	0.0254 (4)	0.0259 (4)	−0.0063 (4)	−0.0116 (4)	−0.0009 (3)
C9	0.0385 (5)	0.0249 (4)	0.0240 (4)	−0.0057 (3)	−0.0091 (3)	−0.0028 (3)
C10	0.0389 (5)	0.0254 (4)	0.0381 (5)	−0.0054 (4)	−0.0111 (4)	−0.0034 (4)
C11	0.0556 (7)	0.0410 (6)	0.0608 (8)	−0.0076 (5)	−0.0281 (6)	−0.0112 (5)
C12	0.0610 (9)	0.0441 (7)	0.1198 (16)	−0.0066 (6)	−0.0511 (10)	−0.0159 (8)
C13	0.0406 (7)	0.0385 (7)	0.1285 (17)	−0.0077 (5)	−0.0178 (9)	−0.0063 (8)
C14	0.0456 (7)	0.0411 (7)	0.0778 (10)	−0.0069 (5)	0.0034 (7)	0.0027 (6)
C15	0.0454 (6)	0.0376 (6)	0.0426 (6)	−0.0069 (5)	−0.0042 (5)	−0.0013 (4)
C16	0.0414 (5)	0.0350 (5)	0.0313 (5)	−0.0071 (4)	−0.0097 (4)	0.0036 (4)
C17	0.0566 (7)	0.0426 (6)	0.0510 (7)	0.0028 (5)	−0.0171 (6)	0.0076 (5)

Geometric parameters (Å, º)

O1—C7	1.2261 (13)	C7—C8	1.5297 (16)
O2—C8	1.4046 (13)	C8—C9	1.5382 (13)
O2—H2	0.83 (2)	C8—H8	0.9800

O3—C16	1.2247 (14)	C9—C10	1.5148 (14)
N1—C7	1.3510 (15)	C9—H9	0.9800
N1—C1	1.4149 (14)	C10—C11	1.3897 (17)
N1—H1	0.89 (2)	C10—C15	1.3905 (16)
N2—C16	1.3637 (14)	C11—C12	1.395 (2)
N2—C6	1.4302 (13)	C11—H11	0.9300
N2—C9	1.4776 (12)	C12—C13	1.378 (3)
C1—C2	1.3886 (17)	C12—H12	0.9300
C1—C6	1.3950 (15)	C13—C14	1.375 (3)
C2—C3	1.381 (2)	C13—H13	0.9300
C2—H2A	0.9300	C14—C15	1.3818 (19)
C3—C4	1.378 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.3814 (19)	C16—C17	1.5031 (17)
C4—H4	0.9300	C17—H17A	0.9600
C5—C6	1.3897 (15)	C17—H17B	0.9600
C5—H5	0.9300	C17—H17C	0.9600

C8—O2—H2	109.8 (14)	N2—C9—C10	111.43 (8)
C7—N1—C1	124.00 (10)	N2—C9—C8	109.66 (8)
C7—N1—H1	114.4 (12)	C10—C9—C8	113.46 (9)
C1—N1—H1	120.4 (12)	N2—C9—H9	107.3
C16—N2—C6	122.88 (9)	C10—C9—H9	107.3
C16—N2—C9	118.55 (8)	C8—C9—H9	107.3
C6—N2—C9	118.41 (8)	C11—C10—C15	119.09 (12)
C2—C1—C6	119.01 (11)	C11—C10—C9	122.52 (10)
C2—C1—N1	120.58 (11)	C15—C10—C9	118.38 (10)
C6—C1—N1	120.40 (10)	C10—C11—C12	119.33 (15)
C3—C2—C1	120.25 (13)	C10—C11—H11	120.3
C3—C2—H2A	119.9	C12—C11—H11	120.3
C1—C2—H2A	119.9	C13—C12—C11	120.92 (16)
C4—C3—C2	120.84 (13)	C13—C12—H12	119.5
C4—C3—H3	119.6	C11—C12—H12	119.5
C2—C3—H3	119.6	C14—C13—C12	119.71 (14)
C3—C4—C5	119.44 (13)	C14—C13—H13	120.1
C3—C4—H4	120.3	C12—C13—H13	120.1
C5—C4—H4	120.3	C13—C14—C15	120.03 (16)
C4—C5—C6	120.37 (12)	C13—C14—H14	120.0
C4—C5—H5	119.8	C15—C14—H14	120.0
C6—C5—H5	119.8	C14—C15—C10	120.91 (14)
C5—C6—C1	120.05 (10)	C14—C15—H15	119.5
C5—C6—N2	121.01 (10)	C10—C15—H15	119.5
C1—C6—N2	118.94 (9)	O3—C16—N2	120.90 (10)
O1—C7—N1	122.08 (11)	O3—C16—C17	121.05 (11)
O1—C7—C8	121.49 (10)	N2—C16—C17	118.04 (10)
N1—C7—C8	116.36 (9)	C16—C17—H17A	109.5
O2—C8—C7	111.68 (8)	C16—C17—H17B	109.5
O2—C8—C9	110.91 (8)	H17A—C17—H17B	109.5

C7—C8—C9	111.53 (9)	C16—C17—H17C	109.5
O2—C8—H8	107.5	H17A—C17—H17C	109.5
C7—C8—H8	107.5	H17B—C17—H17C	109.5
C9—C8—H8	107.5		

C7—N1—C1—C2	−134.65 (15)	C6—N2—C9—C10	−87.10 (10)
C7—N1—C1—C6	45.87 (19)	C16—N2—C9—C8	−145.05 (10)
C6—C1—C2—C3	−1.6 (3)	C6—N2—C9—C8	39.37 (12)
N1—C1—C2—C3	178.87 (17)	O2—C8—C9—N2	173.40 (8)
C1—C2—C3—C4	0.3 (3)	C7—C8—C9—N2	48.24 (11)
C2—C3—C4—C5	0.1 (3)	O2—C8—C9—C10	−61.29 (11)
C3—C4—C5—C6	0.7 (3)	C7—C8—C9—C10	173.56 (8)
C4—C5—C6—C1	−2.0 (2)	N2—C9—C10—C11	84.57 (12)
C4—C5—C6—N2	177.86 (14)	C8—C9—C10—C11	−39.79 (14)
C2—C1—C6—C5	2.5 (2)	N2—C9—C10—C15	−94.64 (11)
N1—C1—C6—C5	−178.04 (12)	C8—C9—C10—C15	140.99 (10)
C2—C1—C6—N2	−177.42 (13)	C15—C10—C11—C12	0.26 (18)
N1—C1—C6—N2	2.06 (18)	C9—C10—C11—C12	−178.95 (11)
C16—N2—C6—C5	−67.48 (16)	C10—C11—C12—C13	0.0 (2)
C9—N2—C6—C5	107.89 (12)	C11—C12—C13—C14	−0.5 (2)
C16—N2—C6—C1	112.42 (13)	C12—C13—C14—C15	0.7 (2)
C9—N2—C6—C1	−72.21 (14)	C13—C14—C15—C10	−0.4 (2)
C1—N1—C7—O1	−178.75 (11)	C11—C10—C15—C14	−0.10 (18)
C1—N1—C7—C8	4.08 (17)	C9—C10—C15—C14	179.14 (11)
O1—C7—C8—O2	−18.12 (14)	C6—N2—C16—O3	175.01 (10)
N1—C7—C8—O2	159.07 (10)	C9—N2—C16—O3	−0.35 (16)
O1—C7—C8—C9	106.61 (11)	C6—N2—C16—C17	−6.10 (16)
N1—C7—C8—C9	−76.20 (12)	C9—N2—C16—C17	178.53 (10)
C16—N2—C9—C10	88.48 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (2)	2.04 (2)	2.924 (1)	175 (2)
O2—H2···O3^{ii}	0.83 (2)	2.09 (2)	2.905 (1)	168 (2)

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