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(3*R*,4*Z*)-1,3-Diethyl-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one

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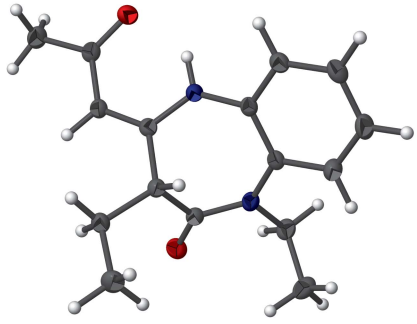
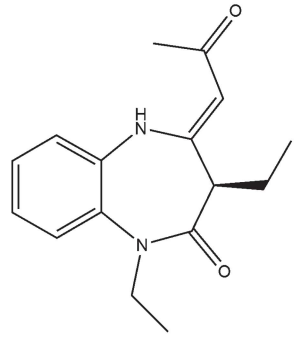
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{20}N_2O_2$, the seven-membered ring adopts a bowl-shaped conformation while the orientation of the 2-oxopropylidene substituent is determined by an intramolecular N—H···O hydrogen bond, which generates an $S(6)$ ring. In the crystal, inversion dimers linked by pairs of very weak C—H···O interactions occur, which generate $R_2^2(8)$ loops.

3D view



Chemical scheme



Structure description

1,4- and 1,5-Benzodiazepines are commonly used as anxiolytic and anticonvulsive drugs: these effects are primarily mediated *via* the benzodiazepine receptors located in the central nervous system (Tallman *et al.*, 1980). In a continuation on our studies (Sebhaoui *et al.*, 2017; Samba *et al.*, 2018) of benzodiazepine derivatives, we report here the alkylation of (4*Z*)-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1,5-benzodiazepin-2-one with ethyl iodide under solid–liquid phase-transfer catalytic conditions leading to the title compound.

The seven-membered ring adopts a bowl-shaped conformation with puckering parameters $Q(2)$ = 0.8767 (13) Å, $Q(3)$ = 0.2633 (13) Å, $\varphi(2)$ = 203.90 (8)° and $\varphi(3)$ = 309.7 (3)°. The total puckering is 0.9154 (13) Å. The orientation of the 2-oxopropylidene substituent is largely determined by the intramolecular N1—H1···O2 hydrogen bond (Table 1 and Fig. 1). Apart from a possible weak C12—H12*A*···O2 hydrogen bond (H···O is 0.09 Å less than the sum of the van der Walls' radii), the packing is determined by van der Waals contacts (Fig. 2).

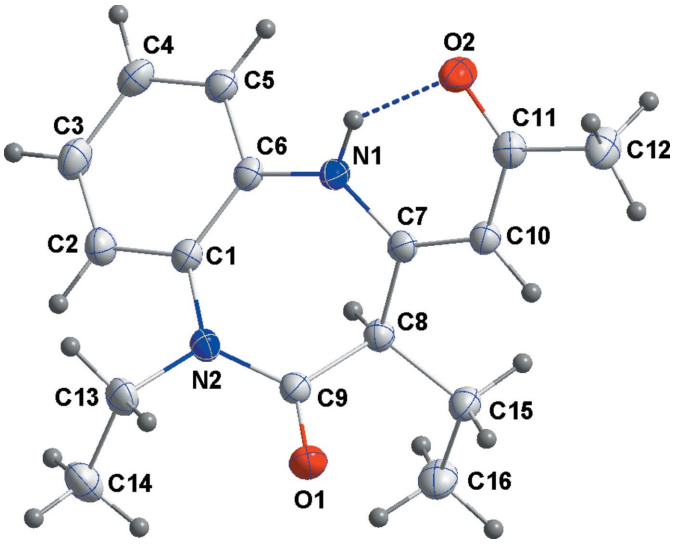


Figure 1
The title molecule with 50% probability ellipsoids.

Synthesis and crystallization

To a solution of (4*Z*)-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1,5-benzodiazepin-2-one (1 mmol) and potassium carbonate K_2CO_3 (1.5 mmol) in 30 ml of *N*,*N*-dimethylformamide, were added ethyl iodide (0.02 mol) and tetra-*n*-butylammonium bromide as a phase-transfer catalyst. The reaction mixture was stirred at room temperature for 10 h. The residue obtained, after evaporation of the solvent, was chromatographed on a silica gel column using a hexane/ethyl acetate 4:1 mixture as eluent. The solid obtained was recrystallized from ethanol solution to afford colourless plates of the title compound.

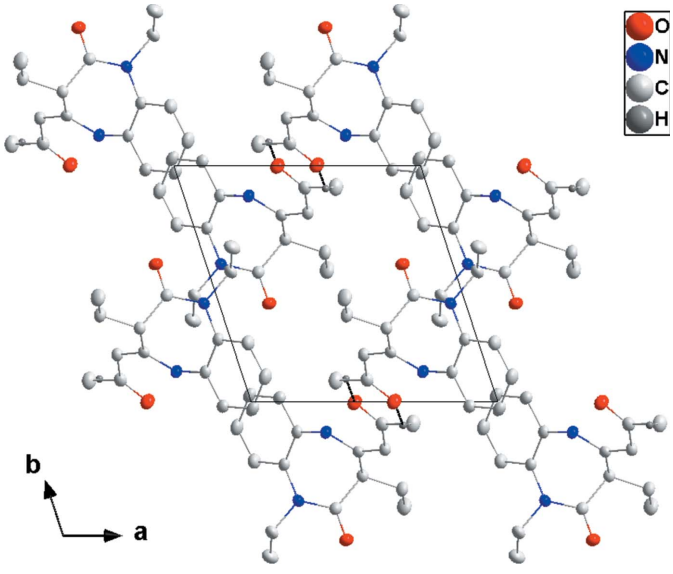


Figure 2
Packing viewed along the *c*-axis direction.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.887 (19)	1.897 (19)	2.6326 (13)	139.2 (16)
C12—H12*A*···O2^{i}	0.98 (2)	2.63 (2)	3.5334 (17)	153.4 (17)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{20}N_2O_2$
M_r	272.34
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, *b*, *c* (Å)	8.8546 (2), 8.9841 (2), 9.5523 (2)
α, β, γ (°)	98.481 (1), 96.412 (1), 106.772 (1)
V (Å^3)	710.00 (3)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	0.68
Crystal size (mm)	0.31 × 0.15 × 0.07
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.88, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5416, 2616, 2387
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.037, 0.091, 1.05
No. of reflections	2616
No. of parameters	262
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.22

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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full crystallographic data

IUCrData (2018). **3**, x180515 [https://doi.org/10.1107/S2414314618005151]

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Crystal data

$C_{16}H_{20}N_2O_2$
M_r = 272.34
Triclinic, $P\overline{1}$
a = 8.8546 (2) Å
b = 8.9841 (2) Å
c = 9.5523 (2) Å
α = 98.481 (1)°
β = 96.412 (1)°
γ = 106.772 (1)°
V = 710.00 (3) Å^3
Z = 2
F(000) = 292
D_x = 1.274 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 4659 reflections
θ = 5.2–72.4°
μ = 0.68 mm^{-1}
T = 150 K
Plate, colourless
0.31 × 0.15 × 0.07 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.88, T_{max} = 0.96
5416 measured reflections
2616 independent reflections
2387 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 72.4°, θ_{min} = 5.2°
h = −10→10
k = −11→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.091
S = 1.05
2616 reflections
262 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 0.2722P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL2018* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0107 (11)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.20343 (11)	0.41685 (11)	0.35492 (10)	0.0288 (2)
O2	0.41944 (11)	0.99801 (11)	0.76940 (9)	0.0286 (2)
N1	0.26316 (12)	0.87183 (13)	0.50781 (11)	0.0207 (2)
H1	0.282 (2)	0.940 (2)	0.590 (2)	0.040 (5)*
N2	0.06087 (12)	0.58264 (12)	0.31111 (11)	0.0216 (2)
C1	0.05091 (14)	0.73584 (15)	0.29983 (13)	0.0206 (3)
C2	−0.06584 (15)	0.74986 (17)	0.19536 (14)	0.0272 (3)
H2	−0.131 (2)	0.655 (2)	0.1266 (18)	0.036 (4)*
C3	−0.09068 (16)	0.89355 (17)	0.18786 (15)	0.0301 (3)
H3	−0.175 (2)	0.902 (2)	0.1165 (19)	0.042 (5)*
C4	0.00296 (16)	1.02877 (16)	0.28445 (15)	0.0272 (3)
H4	−0.0131 (19)	1.1329 (19)	0.2803 (17)	0.031 (4)*
C5	0.12020 (15)	1.01790 (15)	0.38658 (14)	0.0231 (3)
H5	0.1877 (19)	1.1120 (19)	0.4583 (17)	0.031 (4)*
C6	0.14635 (14)	0.87331 (15)	0.39597 (12)	0.0196 (3)
C7	0.36913 (13)	0.78969 (14)	0.49951 (13)	0.0192 (3)
C8	0.35391 (14)	0.68791 (15)	0.35474 (13)	0.0203 (3)
H8	0.3357 (17)	0.7520 (17)	0.2802 (16)	0.022 (3)*
C9	0.20103 (14)	0.54911 (15)	0.33985 (13)	0.0211 (3)
C10	0.48033 (14)	0.79991 (15)	0.61585 (13)	0.0213 (3)
H10	0.5525 (18)	0.7358 (17)	0.6064 (16)	0.023 (4)*
C11	0.50091 (14)	0.90620 (15)	0.74916 (13)	0.0225 (3)
C12	0.62834 (17)	0.9093 (2)	0.86900 (15)	0.0311 (3)
H12A	0.585 (3)	0.902 (2)	0.958 (2)	0.059 (6)*
H12B	0.713 (3)	1.014 (3)	0.883 (2)	0.071 (7)*
H12C	0.680 (2)	0.828 (2)	0.8474 (19)	0.042 (5)*
C13	−0.08886 (15)	0.44886 (16)	0.28977 (15)	0.0261 (3)
H13A	−0.175 (2)	0.4960 (19)	0.3099 (17)	0.031 (4)*
H13B	−0.0722 (18)	0.3840 (19)	0.3624 (17)	0.028 (4)*
C14	−0.12777 (18)	0.34639 (18)	0.14073 (16)	0.0338 (3)
H14A	−0.221 (2)	0.253 (2)	0.136 (2)	0.050 (5)*
H14B	−0.155 (2)	0.406 (2)	0.067 (2)	0.051 (5)*
H14C	−0.034 (2)	0.3101 (19)	0.1212 (17)	0.034 (4)*
C15	0.49743 (15)	0.63217 (16)	0.32949 (13)	0.0245 (3)
H15A	0.5090 (17)	0.5527 (18)	0.3921 (16)	0.024 (4)*

H15B	0.598 (2)	0.7274 (19)	0.3576 (17)	0.030 (4)*
C16	0.48269 (18)	0.5586 (2)	0.17249 (15)	0.0331 (3)
H16A	0.380 (2)	0.468 (2)	0.1413 (19)	0.044 (5)*
H16B	0.574 (2)	0.512 (2)	0.157 (2)	0.046 (5)*
H16C	0.479 (2)	0.638 (2)	0.111 (2)	0.050 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0290 (5)	0.0216 (5)	0.0362 (5)	0.0095 (4)	0.0027 (4)	0.0058 (4)
O2	0.0321 (5)	0.0323 (5)	0.0228 (4)	0.0156 (4)	0.0017 (4)	0.0006 (4)
N1	0.0210 (5)	0.0230 (5)	0.0181 (5)	0.0095 (4)	0.0002 (4)	0.0011 (4)
N2	0.0179 (5)	0.0203 (5)	0.0246 (5)	0.0040 (4)	0.0015 (4)	0.0037 (4)
C1	0.0188 (5)	0.0227 (6)	0.0217 (6)	0.0081 (5)	0.0039 (4)	0.0049 (5)
C2	0.0234 (6)	0.0307 (7)	0.0260 (6)	0.0093 (5)	−0.0015 (5)	0.0028 (6)
C3	0.0271 (6)	0.0376 (8)	0.0289 (7)	0.0159 (6)	−0.0005 (5)	0.0085 (6)
C4	0.0279 (6)	0.0289 (7)	0.0309 (7)	0.0152 (5)	0.0073 (5)	0.0099 (6)
C5	0.0218 (6)	0.0240 (6)	0.0251 (6)	0.0089 (5)	0.0058 (5)	0.0044 (5)
C6	0.0179 (5)	0.0238 (6)	0.0189 (6)	0.0081 (5)	0.0044 (4)	0.0048 (5)
C7	0.0178 (5)	0.0187 (6)	0.0210 (6)	0.0047 (4)	0.0046 (4)	0.0044 (5)
C8	0.0190 (6)	0.0224 (6)	0.0192 (6)	0.0067 (5)	0.0025 (4)	0.0031 (5)
C9	0.0212 (6)	0.0224 (6)	0.0192 (6)	0.0075 (5)	0.0017 (4)	0.0019 (5)
C10	0.0194 (6)	0.0238 (6)	0.0211 (6)	0.0077 (5)	0.0027 (5)	0.0040 (5)
C11	0.0215 (6)	0.0259 (6)	0.0202 (6)	0.0065 (5)	0.0039 (5)	0.0063 (5)
C12	0.0305 (7)	0.0427 (8)	0.0207 (6)	0.0151 (6)	−0.0002 (5)	0.0032 (6)
C13	0.0197 (6)	0.0242 (6)	0.0314 (7)	0.0020 (5)	0.0039 (5)	0.0057 (6)
C14	0.0318 (7)	0.0271 (7)	0.0341 (8)	0.0014 (6)	−0.0035 (6)	0.0022 (6)
C15	0.0221 (6)	0.0300 (7)	0.0225 (6)	0.0114 (5)	0.0031 (5)	0.0017 (6)
C16	0.0311 (7)	0.0428 (9)	0.0259 (7)	0.0154 (7)	0.0066 (6)	−0.0016 (6)

Geometric parameters (Å, º)

O1—C9	1.2237 (15)	C8—C9	1.5291 (17)
O2—C11	1.2513 (15)	C8—H8	1.007 (15)
N1—C7	1.3529 (15)	C10—C11	1.4338 (17)
N1—C6	1.4059 (15)	C10—H10	0.978 (15)
N1—H1	0.887 (19)	C11—C12	1.5061 (17)
N2—C9	1.3676 (15)	C12—H12A	0.98 (2)
N2—C1	1.4232 (15)	C12—H12B	1.00 (3)
N2—C13	1.4804 (15)	C12—H12C	0.980 (19)
C1—C2	1.4008 (17)	C13—C14	1.5212 (19)
C1—C6	1.4045 (17)	C13—H13A	0.993 (16)
C2—C3	1.3811 (19)	C13—H13B	0.994 (16)
C2—H2	0.978 (18)	C14—H14A	0.99 (2)
C3—C4	1.390 (2)	C14—H14B	1.00 (2)
C3—H3	0.979 (18)	C14—H14C	0.999 (17)
C4—C5	1.3781 (18)	C15—C16	1.5222 (18)
C4—H4	0.991 (16)	C15—H15A	1.016 (15)

C5—C6	1.3973 (17)	C15—H15B	1.017 (17)
C5—H5	0.993 (17)	C16—H16A	1.01 (2)
C7—C10	1.3734 (17)	C16—H16B	1.025 (19)
C7—C8	1.5103 (16)	C16—H16C	0.99 (2)
C8—C15	1.5245 (16)		

C7—N1—C6	126.56 (10)	C7—C10—C11	122.91 (11)
C7—N1—H1	113.3 (12)	C7—C10—H10	118.9 (9)
C6—N1—H1	119.5 (11)	C11—C10—H10	118.2 (9)
C9—N2—C1	124.31 (10)	O2—C11—C10	122.89 (11)
C9—N2—C13	116.88 (10)	O2—C11—C12	118.70 (11)
C1—N2—C13	118.80 (10)	C10—C11—C12	118.40 (11)
C2—C1—C6	118.27 (12)	C11—C12—H12A	110.3 (13)
C2—C1—N2	119.06 (11)	C11—C12—H12B	107.1 (13)
C6—C1—N2	122.52 (10)	H12A—C12—H12B	107.8 (18)
C3—C2—C1	121.58 (12)	C11—C12—H12C	113.4 (11)
C3—C2—H2	119.7 (10)	H12A—C12—H12C	110.9 (16)
C1—C2—H2	118.8 (10)	H12B—C12—H12C	107.0 (17)
C2—C3—C4	119.82 (12)	N2—C13—C14	113.09 (11)
C2—C3—H3	121.0 (10)	N2—C13—H13A	106.4 (9)
C4—C3—H3	119.2 (10)	C14—C13—H13A	111.7 (9)
C5—C4—C3	119.49 (12)	N2—C13—H13B	105.9 (9)
C5—C4—H4	119.7 (9)	C14—C13—H13B	109.3 (9)
C3—C4—H4	120.8 (9)	H13A—C13—H13B	110.3 (13)
C4—C5—C6	121.39 (12)	C13—C14—H14A	109.5 (11)
C4—C5—H5	121.4 (9)	C13—C14—H14B	110.6 (11)
C6—C5—H5	117.2 (9)	H14A—C14—H14B	107.8 (16)
C5—C6—C1	119.44 (11)	C13—C14—H14C	109.2 (10)
C5—C6—N1	117.53 (11)	H14A—C14—H14C	108.8 (14)
C1—C6—N1	122.94 (11)	H14B—C14—H14C	111.0 (15)
N1—C7—C10	121.37 (11)	C16—C15—C8	110.84 (10)
N1—C7—C8	114.87 (10)	C16—C15—H15A	109.6 (8)
C10—C7—C8	123.76 (11)	C8—C15—H15A	111.1 (8)
C7—C8—C15	115.57 (10)	C16—C15—H15B	109.4 (9)
C7—C8—C9	105.80 (9)	C8—C15—H15B	108.3 (9)
C15—C8—C9	111.94 (10)	H15A—C15—H15B	107.5 (12)
C7—C8—H8	107.0 (8)	C15—C16—H16A	110.4 (10)
C15—C8—H8	108.7 (8)	C15—C16—H16B	111.0 (10)
C9—C8—H8	107.4 (8)	H16A—C16—H16B	106.7 (14)
O1—C9—N2	121.94 (11)	C15—C16—H16C	110.0 (11)
O1—C9—C8	122.29 (11)	H16A—C16—H16C	106.9 (15)
N2—C9—C8	115.77 (10)	H16B—C16—H16C	111.7 (15)

C9—N2—C1—C2	141.65 (12)	C10—C7—C8—C15	−16.89 (18)
C13—N2—C1—C2	−37.92 (16)	N1—C7—C8—C9	−72.16 (13)
C9—N2—C1—C6	−42.92 (17)	C10—C7—C8—C9	107.58 (13)
C13—N2—C1—C6	137.50 (12)	C1—N2—C9—O1	176.65 (11)
C6—C1—C2—C3	−1.59 (19)	C13—N2—C9—O1	−3.77 (17)

N2—C1—C2—C3	174.03 (12)	C1—N2—C9—C8	−2.22 (16)
C1—C2—C3—C4	0.8 (2)	C13—N2—C9—C8	177.36 (10)
C2—C3—C4—C5	0.2 (2)	C7—C8—C9—O1	−103.76 (13)
C3—C4—C5—C6	−0.50 (19)	C15—C8—C9—O1	22.94 (16)
C4—C5—C6—C1	−0.26 (18)	C7—C8—C9—N2	75.10 (13)
C4—C5—C6—N1	−176.96 (11)	C15—C8—C9—N2	−158.19 (10)
C2—C1—C6—C5	1.28 (17)	N1—C7—C10—C11	−5.14 (19)
N2—C1—C6—C5	−174.19 (11)	C8—C7—C10—C11	175.13 (11)
C2—C1—C6—N1	177.79 (11)	C7—C10—C11—O2	−0.2 (2)
N2—C1—C6—N1	2.32 (18)	C7—C10—C11—C12	−179.09 (12)
C7—N1—C6—C5	−140.34 (12)	C9—N2—C13—C14	−79.55 (14)
C7—N1—C6—C1	43.09 (18)	C1—N2—C13—C14	100.05 (13)
C6—N1—C7—C10	178.73 (12)	C7—C8—C15—C16	−169.08 (11)
C6—N1—C7—C8	−1.52 (17)	C9—C8—C15—C16	69.70 (14)
N1—C7—C8—C15	163.37 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.887 (19)	1.897 (19)	2.6326 (13)	139.2 (16)
C12—H12*A*···O2^{i}	0.98 (2)	2.63 (2)	3.5334 (17)	153.4 (17)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+2.