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4-(2-Oxopropyl)-1,3-bis(prop-2-yn-1-yl)-2,3,4,5-tetrahydro-1,5-benzodiazepin-2(1*H*)-one

Jihad Sebhaoui,[a]* Youness El Bakri,[a] Ibtissam Rayni,[a] El Mokhtar Essassi[a] and Joel T. Mague[b]

[a]Laboratoire de Chimie Organique Hétérocyclique, URAC 21, Pôle de Compétence Pharmacochimie, Av Ibn Battouta, BP 1014, Faculté des Sciences, Mohammed V University, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: sebhaoui.jihad@gmail.com

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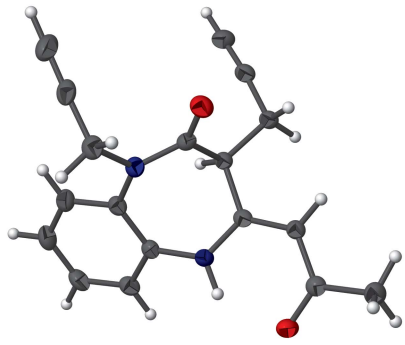
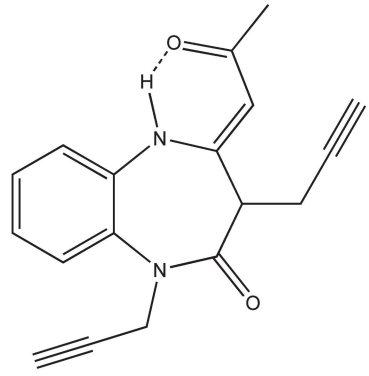
Structural data: full structural data are available from iucrdata.iucr.org

In the title molecule, $C_{18}H_{16}N_2O_2$, the seven-membered diazepinone ring adopts a boat conformation. An intramolecular N—H···O hydrogen bond encloses an *S*(6) ring. The two propynyl substituents each point away from the same face of the benzodiazepinone ring system. In the crystal, C—H···O hydrogen bonds form double chains of molecules along the *c*-axis direction.

3D view



Chemical scheme



Structure description

Benzodiazepine derivatives have been the object of intense investigation in medicinal chemistry because of their remarkable ability to depress activity in the central nervous system and are now one of the most widely prescribed class of psychotropic drugs (Zellou *et al.*, 1998, 1999; Rudolph *et al.*, 1999). The area of biological interest of 1,5-benzodiazepine derivatives has been extended to include antibiotics (Knabe *et al.*, 1995) and the treatment of various diseases such as cancer (Atwal *et al.*, 1987), viral infection (HIV) (Di Braccio *et al.*, 2001) and cardiovascular disorders (Claremon *et al.*, 1998

The conformation of the title molecule is partially determined by an intramolecular N2—H2*A*···O2 hydrogen bond (Table 1 and Fig. 1). The heterocyclic ring adopts a boat conformation with puckering parameters $Q(2)$ = 0.899 (1) Å, $\varphi(2)$ = 206.26 (8)°, $Q(3)$ = 0.261 (1) Å and $\varphi(3)$ = 303.1 (3)°. The two propynyl substituents, C14—C15—H15 and C17—C18—H18 each point away from the same face of the benzodiazepinone ring system and one of these is involved in C18—H18···O2^{i} hydrogen bonds that form $C(10)$ chains along *c*. Additional weaker C12—H12*A*···O1^{ii} contacts form inversion dimers, enclosing $R_2^2(16)$ rings, and these link adjacent chains to produce a double chain propagating along *c*, Fig. 2.

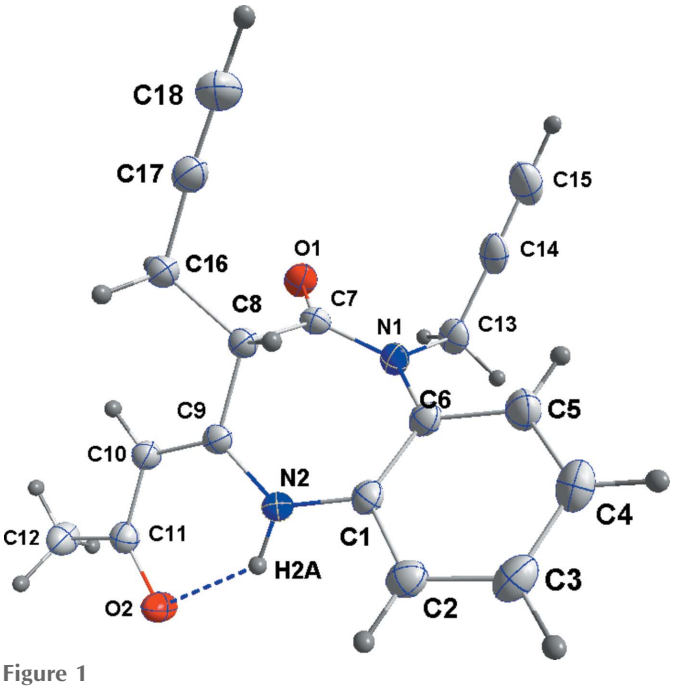


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids. The intramolecular hydrogen bond is shown as a dotted line.

Synthesis and crystallization

To a solution of (4*E*)-2-oxopropylidene-1,5-benzodiazepin-2-one (0.01 mol, 2.16 g) in *N*,*N*-dimethylformamid (60 ml), was added K_2CO_3 (0.02 mol, 2.76 g), propargyl bromide (0.02 mol, 5,84 g) and tetra *n*-butylammonium bromide (0.001 mol, 0.321 g). The reaction mixture was stirred at room temperature for 48 h. The solution was filtered and the solvent was removed under reduced pressure. The residue was chromatographed on a silica-gel column using hexane and ethyl acetate (80/20) as eluents. Recrystallization from this solution gave the title compound as a white crystals suitable for X-ray investigation.

Refinement

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

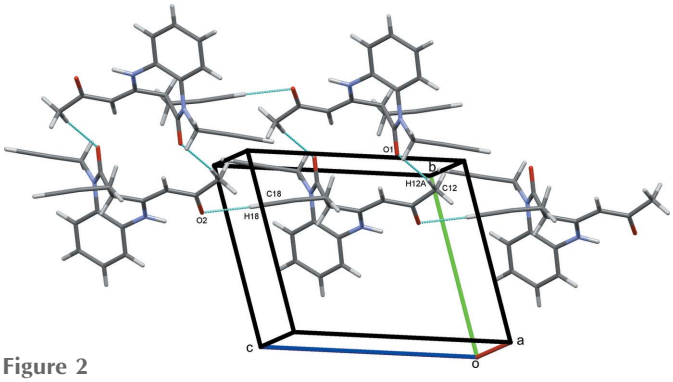


Figure 2
Packing viewed along the *a* axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O2	0.939 (18)	1.918 (18)	2.6678 (13)	135.2 (14)
C18—H18···O2^{i}	0.95	2.35	3.3012 (16)	178
C12—H12*A*···O1^{ii}	0.98	2.70	3.664 (2)	168

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{16}N_2O_2$
M_r	292.33
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	8.6905 (2), 9.0249 (3), 10.8240 (3)
α, β, γ (°)	68.803 (1), 78.520 (1), 72.980 (1)
V (Å^3)	752.69 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.69
Crystal size (mm)	0.17 × 0.13 × 0.13
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.82, 0.92
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5877, 2802, 2554
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.107, 1.05
No. of reflections	2802
No. of parameters	205
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.22

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012), *Mercury* (Macrae *et al.*, 2008) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

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4-(2-Oxopropylidene)-1,3-bis(prop-2-yn-1-yl)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one

Crystal data

$C_{18}H_{16}N_2O_2$
M_r = 292.33
Triclinic, $P\overline{1}$
a = 8.6905 (2) Å
b = 9.0249 (3) Å
c = 10.8240 (3) Å
α = 68.803 (1)°
β = 78.520 (1)°
γ = 72.980 (1)°
V = 752.69 (4) Å^3
Z = 2
$F(000)$ = 308
D_x = 1.290 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5019 reflections
θ = 5.4–72.4°
μ = 0.69 mm^{-1}
T = 150 K
Block, colourless
0.17 × 0.13 × 0.13 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*, Bruker, 2016)
T_{min} = 0.82, T_{max} = 0.92
5877 measured reflections
2802 independent reflections
2554 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 72.4°, θ_{min} = 5.4°
h = −10→10
k = −11→11
l = −12→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.107
S = 1.05
2802 reflections
205 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.166P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0185 (19)

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.29131 (10)	−0.00462 (10)	0.35510 (9)	0.0299 (2)
O2	0.08148 (11)	0.34999 (11)	0.72733 (8)	0.0283 (2)
H2A	0.225 (2)	0.395 (2)	0.5665 (18)	0.041 (4)*
N1	0.44188 (11)	0.17555 (12)	0.32357 (10)	0.0235 (2)
N2	0.24209 (12)	0.37133 (12)	0.48668 (10)	0.0222 (2)
C1	0.36309 (13)	0.42678 (14)	0.38710 (12)	0.0220 (3)
C2	0.39430 (14)	0.57554 (15)	0.37361 (13)	0.0265 (3)
H2	0.3329	0.6365	0.4301	0.032*
C3	0.51333 (16)	0.63519 (16)	0.27926 (14)	0.0313 (3)
H3	0.5339	0.7361	0.2716	0.038*
C4	0.60277 (16)	0.54749 (17)	0.19572 (14)	0.0345 (3)
H4	0.6834	0.5892	0.1298	0.041*
C5	0.57435 (15)	0.39900 (17)	0.20856 (14)	0.0305 (3)
H5	0.6356	0.3396	0.1509	0.037*
C6	0.45668 (14)	0.33579 (14)	0.30532 (12)	0.0232 (3)
C7	0.29822 (14)	0.13457 (14)	0.33893 (11)	0.0221 (3)
C8	0.14850 (13)	0.27376 (14)	0.34124 (12)	0.0210 (3)
H8	0.1702	0.3757	0.2708	0.025*
C9	0.13465 (13)	0.29586 (13)	0.47525 (12)	0.0205 (3)
C10	0.02525 (13)	0.23893 (14)	0.57948 (12)	0.0224 (3)
H10	−0.0429	0.1812	0.5672	0.027*
C11	0.00868 (14)	0.26233 (14)	0.70643 (12)	0.0235 (3)
C12	−0.10022 (16)	0.17527 (17)	0.81733 (13)	0.0311 (3)
H12A	−0.1656	0.1320	0.7809	0.047*
H12B	−0.1717	0.2522	0.8611	0.047*
H12C	−0.0345	0.0848	0.8824	0.047*
C13	0.59039 (15)	0.04794 (15)	0.31350 (14)	0.0289 (3)
H13A	0.6825	0.0767	0.3329	0.035*
H13B	0.5781	−0.0576	0.3814	0.035*
C14	0.62650 (15)	0.02832 (15)	0.18111 (14)	0.0305 (3)
C15	0.65303 (19)	0.00942 (19)	0.07573 (16)	0.0421 (4)
H15	0.6744	−0.0058	−0.0090	0.050*
C16	−0.00053 (14)	0.24122 (15)	0.31059 (12)	0.0249 (3)
H16A	−0.0093	0.1284	0.3643	0.030*

H16B	−0.0994	0.3185	0.3341	0.030*
C17	0.01388 (14)	0.26199 (15)	0.16813 (13)	0.0271 (3)
C18	0.03206 (17)	0.28788 (17)	0.05105 (14)	0.0336 (3)
H18	0.0466	0.3086	−0.0425	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0299 (5)	0.0217 (4)	0.0404 (5)	−0.0055 (3)	−0.0037 (4)	−0.0135 (4)
O2	0.0341 (5)	0.0294 (5)	0.0250 (5)	−0.0108 (4)	−0.0018 (4)	−0.0113 (4)
N1	0.0198 (5)	0.0203 (5)	0.0298 (5)	−0.0021 (4)	−0.0012 (4)	−0.0104 (4)
N2	0.0231 (5)	0.0224 (5)	0.0234 (5)	−0.0063 (4)	−0.0021 (4)	−0.0095 (4)
C1	0.0194 (5)	0.0221 (6)	0.0241 (6)	−0.0043 (4)	−0.0048 (4)	−0.0062 (5)
C2	0.0255 (6)	0.0227 (6)	0.0329 (7)	−0.0040 (5)	−0.0076 (5)	−0.0100 (5)
C3	0.0299 (6)	0.0262 (6)	0.0407 (7)	−0.0117 (5)	−0.0080 (5)	−0.0080 (6)
C4	0.0281 (6)	0.0361 (7)	0.0388 (7)	−0.0152 (5)	0.0008 (5)	−0.0079 (6)
C5	0.0241 (6)	0.0327 (7)	0.0350 (7)	−0.0081 (5)	0.0022 (5)	−0.0131 (6)
C6	0.0201 (5)	0.0215 (6)	0.0280 (6)	−0.0042 (4)	−0.0044 (4)	−0.0079 (5)
C7	0.0236 (6)	0.0217 (6)	0.0216 (6)	−0.0042 (4)	−0.0017 (4)	−0.0091 (5)
C8	0.0199 (5)	0.0207 (6)	0.0231 (6)	−0.0039 (4)	−0.0027 (4)	−0.0085 (5)
C9	0.0194 (5)	0.0166 (5)	0.0250 (6)	−0.0001 (4)	−0.0053 (4)	−0.0080 (5)
C10	0.0218 (5)	0.0209 (5)	0.0251 (6)	−0.0047 (4)	−0.0029 (4)	−0.0082 (5)
C11	0.0229 (5)	0.0207 (6)	0.0246 (6)	−0.0019 (4)	−0.0048 (4)	−0.0062 (5)
C12	0.0325 (7)	0.0364 (7)	0.0243 (6)	−0.0128 (5)	−0.0015 (5)	−0.0067 (6)
C13	0.0220 (6)	0.0245 (6)	0.0373 (7)	0.0006 (5)	−0.0034 (5)	−0.0114 (5)
C14	0.0236 (6)	0.0235 (6)	0.0408 (8)	−0.0042 (5)	0.0050 (5)	−0.0118 (6)
C15	0.0430 (8)	0.0423 (8)	0.0427 (9)	−0.0178 (7)	0.0148 (6)	−0.0200 (7)
C16	0.0224 (6)	0.0278 (6)	0.0270 (6)	−0.0053 (5)	−0.0038 (4)	−0.0119 (5)
C17	0.0246 (6)	0.0283 (6)	0.0319 (7)	−0.0050 (5)	−0.0058 (5)	−0.0134 (5)
C18	0.0370 (7)	0.0386 (7)	0.0302 (7)	−0.0101 (6)	−0.0055 (5)	−0.0151 (6)

Geometric parameters (Å, º)

O1—C7	1.2219 (14)	C8—C16	1.5290 (15)
O2—C11	1.2478 (15)	C8—H8	1.0000
N1—C7	1.3662 (15)	C9—C10	1.3713 (17)
N1—C6	1.4280 (15)	C10—C11	1.4376 (16)
N1—C13	1.4726 (15)	C10—H10	0.9500
N2—C9	1.3529 (14)	C11—C12	1.5017 (17)
N2—C1	1.4088 (15)	C12—H12A	0.9800
N2—H2A	0.939 (18)	C12—H12B	0.9800
C1—C2	1.3975 (16)	C12—H12C	0.9800
C1—C6	1.4027 (17)	C13—C14	1.4681 (18)
C2—C3	1.3823 (18)	C13—H13A	0.9900
C2—H2	0.9500	C13—H13B	0.9900
C3—C4	1.388 (2)	C14—C15	1.182 (2)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.3862 (19)	C16—C17	1.4685 (17)

C4—H4	0.9500	C16—H16A	0.9900
C5—C6	1.3969 (17)	C16—H16B	0.9900
C5—H5	0.9500	C17—C18	1.1888 (19)
C7—C8	1.5247 (15)	C18—H18	0.9500
C8—C9	1.5113 (15)		

C7—N1—C6	124.46 (9)	C16—C8—H8	108.0
C7—N1—C13	116.81 (10)	N2—C9—C10	122.14 (10)
C6—N1—C13	118.50 (9)	N2—C9—C8	115.06 (10)
C9—N2—C1	125.92 (10)	C10—C9—C8	122.76 (10)
C9—N2—H2A	114.6 (11)	C9—C10—C11	123.22 (10)
C1—N2—H2A	119.3 (11)	C9—C10—H10	118.4
C2—C1—C6	119.19 (11)	C11—C10—H10	118.4
C2—C1—N2	118.15 (10)	O2—C11—C10	122.72 (11)
C6—C1—N2	122.61 (10)	O2—C11—C12	119.61 (11)
C3—C2—C1	120.91 (11)	C10—C11—C12	117.67 (10)
C3—C2—H2	119.5	C11—C12—H12A	109.5
C1—C2—H2	119.5	C11—C12—H12B	109.5
C2—C3—C4	119.91 (12)	H12A—C12—H12B	109.5
C2—C3—H3	120.0	C11—C12—H12C	109.5
C4—C3—H3	120.0	H12A—C12—H12C	109.5
C5—C4—C3	119.92 (12)	H12B—C12—H12C	109.5
C5—C4—H4	120.0	C14—C13—N1	112.32 (10)
C3—C4—H4	120.0	C14—C13—H13A	109.1
C4—C5—C6	120.72 (12)	N1—C13—H13A	109.1
C4—C5—H5	119.6	C14—C13—H13B	109.1
C6—C5—H5	119.6	N1—C13—H13B	109.1
C5—C6—C1	119.31 (11)	H13A—C13—H13B	107.9
C5—C6—N1	118.84 (11)	C15—C14—C13	178.17 (14)
C1—C6—N1	121.74 (10)	C14—C15—H15	180.0
O1—C7—N1	122.21 (11)	C17—C16—C8	109.32 (10)
O1—C7—C8	123.08 (10)	C17—C16—H16A	109.8
N1—C7—C8	114.65 (10)	C8—C16—H16A	109.8
C9—C8—C7	105.29 (9)	C17—C16—H16B	109.8
C9—C8—C16	115.45 (9)	C8—C16—H16B	109.8
C7—C8—C16	111.90 (9)	H16A—C16—H16B	108.3
C9—C8—H8	108.0	C18—C17—C16	174.84 (13)
C7—C8—H8	108.0	C17—C18—H18	180.0

C9—N2—C1—C2	141.12 (11)	C13—N1—C7—C8	−176.84 (10)
C9—N2—C1—C6	−41.41 (16)	O1—C7—C8—C9	103.58 (12)
C6—C1—C2—C3	1.36 (17)	N1—C7—C8—C9	−73.45 (12)
N2—C1—C2—C3	178.93 (11)	O1—C7—C8—C16	−22.58 (16)
C1—C2—C3—C4	0.50 (19)	N1—C7—C8—C16	160.39 (10)
C2—C3—C4—C5	−1.1 (2)	C1—N2—C9—C10	176.94 (10)
C3—C4—C5—C6	−0.2 (2)	C1—N2—C9—C8	−0.93 (15)
C4—C5—C6—C1	2.10 (19)	C7—C8—C9—N2	74.96 (11)
C4—C5—C6—N1	−174.11 (11)	C16—C8—C9—N2	−161.10 (10)

C2—C1—C6—C5	−2.63 (17)	C7—C8—C9—C10	−102.89 (12)
N2—C1—C6—C5	179.92 (11)	C16—C8—C9—C10	21.05 (15)
C2—C1—C6—N1	173.46 (10)	N2—C9—C10—C11	3.68 (17)
N2—C1—C6—N1	−3.99 (17)	C8—C9—C10—C11	−178.62 (10)
C7—N1—C6—C5	−136.02 (12)	C9—C10—C11—O2	7.22 (18)
C13—N1—C6—C5	38.24 (16)	C9—C10—C11—C12	−172.22 (11)
C7—N1—C6—C1	47.87 (16)	C7—N1—C13—C14	77.54 (14)
C13—N1—C6—C1	−137.87 (12)	C6—N1—C13—C14	−97.15 (13)
C6—N1—C7—O1	−179.55 (11)	C9—C8—C16—C17	166.39 (10)
C13—N1—C7—O1	6.11 (17)	C7—C8—C16—C17	−73.20 (12)
C6—N1—C7—C8	−2.49 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.939 (18)	1.918 (18)	2.6678 (13)	135.2 (14)
C18—H18···O2^{i}	0.95	2.35	3.3012 (16)	178
C12—H12*A*···O1^{ii}	0.98	2.70	3.664 (2)	168

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