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(4*E*)-1-Decyl-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-2-one

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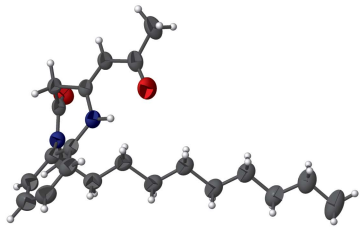
CCDC references: 1494817; 1494817

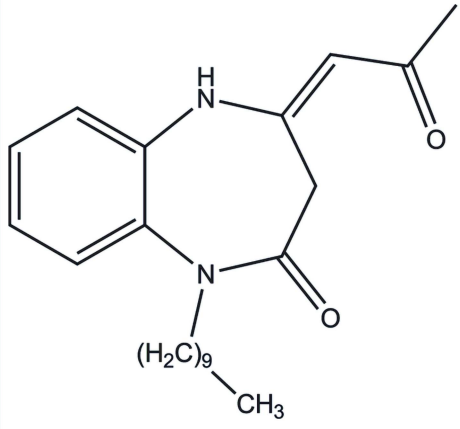
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{22}H_{32}N_2O_2$, forms bilayers with the *n*-decyl chains in extended conformation oriented towards the interior of the bilayer structure. Weak C—H···O interactions help to stabilize the exterior surfaces. The conformation of the seven-membered ring has been analysed.

3D view



Chemical scheme



Structure description

1,5-Benzodiazepine derivatives constitute an important class of nitrogen-containing heterocycles and possess interesting activities as anticonvulsant (Ben-Cherif *et al.*, 2010), antimicrobial (Wang *et al.*, 2015), and anti-inflammatory (Ha *et al.*, 2010) agents. They have also been used as intermediates for the synthesis of benzimidazoles (El Azzaoui *et al.*, 1999) and quinoxalines (Doumbia *et al.*, 2008).

In the crystal, the title compound adopts a U-shaped conformation (Fig. 1). A puckering analysis of the conformation of the seven-membered ring yielded the parameters q_2 = 0.888 (1) Å, φ_2 = 32.74 (8)°, q_3 = 0.203 (1) Å and φ_3 = 127.5 (4)°. An intramolecular N2—H2*A*···O2 hydrogen bond (Table 1) occurs.

In the crystal, the molecules pack to form bilayers with the *n*-decyl chains extended to fill the interior, and with the substituted benzodiazepine units on the surfaces (Fig. 2). Weak C5—H5···O2^{i} contacts [symmetry code (i): $x + 1, y - 1, z$] help to stabilize the hydrophilic portion (Table 1 and Fig. 2).

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*-dimethylformamide (60 ml), was added K_2CO_3 (0.02 mol, 2.76 g), 1-bromodecane

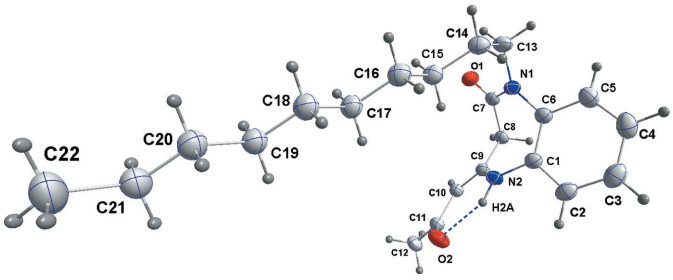


Figure 1
The title molecule with 30% probability ellipsoids for non-H atoms. The dashed line represents an intramolecular hydrogen bond (Table 1, entry 1).

(0.02 mol, 4.42 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 obtained residue was chromatographed on a silica-gel column using a mixture of hexane and ethyl acetate (80/20) as eluent, to afford the title compound as colourless crystals.

Refinement

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

Acknowledgements

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory.

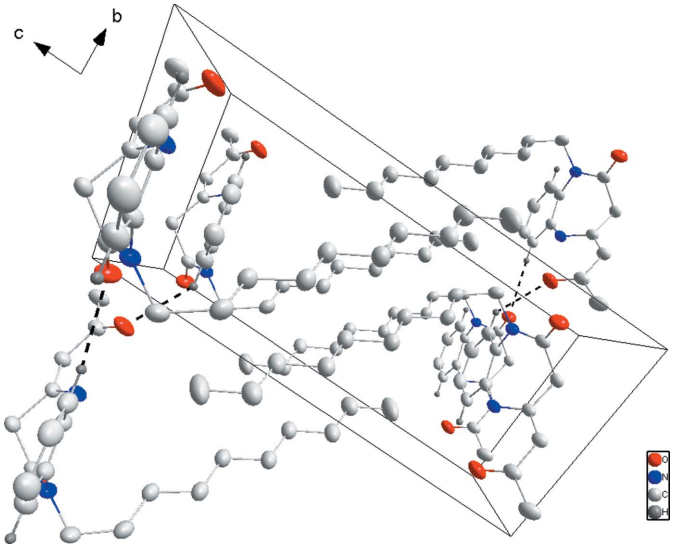


Figure 2
Packing structure viewed along the *a* axis, with C—H···O interactions shown as dashed lines. For clarity only the H atoms involved in these interactions are included.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.887 (16)	1.904 (16)	2.6459 (14)	140.0 (14)
C5—H5···O2^{i}	0.93	2.54	3.3123 (17)	140

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{32}N_2O_2$
M_r	356.49
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, *b*, *c* (Å)	8.4132 (14), 8.4870 (14), 16.670 (3)
α, β, γ (°)	83.560 (2), 85.919 (2), 62.599 (2)
V (Å^3)	1049.8 (3)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.07
Crystal size (mm)	0.44 × 0.32 × 0.26
Data collection	
Diffractometer	Bruker *SMART APEX* CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.85, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20426, 5575, 3821
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.685
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.047, 0.136, 1.04
No. of reflections	5575
No. of parameters	241
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.15

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

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full crystallographic data

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Crystal data

$C_{22}H_{32}N_2O_2$
M_r = 356.49
Triclinic, $P\overline{1}$
a = 8.4132 (14) Å
b = 8.4870 (14) Å
c = 16.670 (3) Å
α = 83.560 (2)°
β = 85.919 (2)°
γ = 62.599 (2)°
V = 1049.8 (3) Å^3
Z = 2
$F(000)$ = 388
D_x = 1.128 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7410 reflections
θ = 2.5–28.9°
μ = 0.07 mm^{-1}
T = 296 K
Block, colourless
0.44 × 0.32 × 0.26 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.85, T_{max} = 0.98
20426 measured reflections
5575 independent reflections
3821 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 29.2°, θ_{min} = 2.5°
h = −11→11
k = −11→11
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.04
5575 reflections
241 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
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.119P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, collected at φ = 0.00, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ, collected at ω = –30.00 and 210.00°. The scan time was 5 sec/frame.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.47356 (13)	0.07555 (12)	0.87693 (7)	0.0643 (3)
O2	0.11670 (14)	0.84072 (12)	0.87720 (7)	0.0745 (3)
N1	0.65022 (13)	0.19922 (12)	0.82244 (6)	0.0450 (2)
N2	0.43528 (14)	0.56271 (13)	0.86512 (6)	0.0466 (3)
H2A	0.360 (2)	0.678 (2)	0.8561 (10)	0.074 (5)*
C1	0.61644 (16)	0.49845 (15)	0.84118 (7)	0.0430 (3)
C2	0.68986 (19)	0.61726 (17)	0.83285 (8)	0.0528 (3)
H2	0.6181	0.7360	0.8420	0.063*
C3	0.8672 (2)	0.5606 (2)	0.81126 (9)	0.0641 (4)
H3	0.9141	0.6415	0.8043	0.077*
C4	0.9758 (2)	0.3830 (2)	0.79987 (10)	0.0686 (4)
H4	1.0970	0.3432	0.7877	0.082*
C5	0.90426 (18)	0.26497 (18)	0.80653 (9)	0.0576 (3)
H5	0.9781	0.1460	0.7985	0.069*
C6	0.72332 (16)	0.32080 (15)	0.82512 (7)	0.0440 (3)
C7	0.53781 (16)	0.17720 (14)	0.88058 (8)	0.0448 (3)
C8	0.49415 (15)	0.29106 (14)	0.95032 (7)	0.0429 (3)
H8A	0.4383	0.2477	0.9942	0.052*
H8B	0.6035	0.2837	0.9699	0.052*
C9	0.36916 (15)	0.48169 (14)	0.92314 (7)	0.0410 (3)
C10	0.20044 (16)	0.56744 (15)	0.95487 (8)	0.0457 (3)
H10	0.1610	0.5038	0.9931	0.055*
C11	0.08004 (17)	0.75103 (16)	0.93245 (9)	0.0530 (3)
C12	−0.09418 (18)	0.83550 (19)	0.97834 (11)	0.0696 (4)
H12A	−0.0899	0.7611	1.0268	0.104*
H12B	−0.1898	0.8492	0.9455	0.104*
H12C	−0.1145	0.9503	0.9921	0.104*
C13	0.69323 (19)	0.09919 (17)	0.75086 (8)	0.0561 (3)
H13A	0.6405	0.0180	0.7577	0.067*
H13B	0.8221	0.0286	0.7460	0.067*
C14	0.62465 (19)	0.22126 (19)	0.67381 (8)	0.0580 (3)
H14A	0.6848	0.2958	0.6653	0.070*
H14B	0.6569	0.1486	0.6287	0.070*
C15	0.42421 (19)	0.34017 (19)	0.67379 (8)	0.0561 (3)
H15A	0.3926	0.4197	0.7162	0.067*
H15B	0.3635	0.2667	0.6860	0.067*
C16	0.3583 (2)	0.4507 (2)	0.59375 (8)	0.0614 (4)
H16A	0.4224	0.5209	0.5812	0.074*
H16B	0.3887	0.3703	0.5518	0.074*
C17	0.1596 (2)	0.5751 (2)	0.59099 (9)	0.0618 (4)

H17A	0.1287	0.6593	0.6313	0.074*
H17B	0.0943	0.5064	0.6047	0.074*
C18	0.1012 (2)	0.6778 (2)	0.50848 (9)	0.0656 (4)
H18A	0.1697	0.7432	0.4946	0.079*
H18B	0.1316	0.5925	0.4687	0.079*
C19	−0.0946 (2)	0.8067 (2)	0.50190 (9)	0.0663 (4)
H19A	−0.1258	0.8926	0.5414	0.080*
H19B	−0.1639	0.7418	0.5151	0.080*
C20	−0.1467 (2)	0.9068 (2)	0.41840 (10)	0.0711 (4)
H20A	−0.0756	0.9696	0.4051	0.085*
H20B	−0.1159	0.8203	0.3792	0.085*
C21	−0.3395 (3)	1.0373 (2)	0.40996 (12)	0.0848 (5)
H21A	−0.3708	1.1237	0.4492	0.102*
H21B	−0.4110	0.9746	0.4227	0.102*
C22	−0.3878 (3)	1.1358 (3)	0.32680 (14)	0.1133 (8)
H22A	−0.5132	1.2178	0.3262	0.170*
H22B	−0.3615	1.0519	0.2876	0.170*
H22C	−0.3194	1.2003	0.3139	0.170*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0662 (6)	0.0505 (5)	0.0877 (7)	−0.0352 (5)	0.0045 (5)	−0.0153 (5)
O2	0.0675 (7)	0.0420 (5)	0.0939 (8)	−0.0118 (5)	−0.0011 (6)	0.0115 (5)
N1	0.0496 (6)	0.0347 (5)	0.0484 (6)	−0.0169 (4)	−0.0028 (4)	−0.0047 (4)
N2	0.0485 (6)	0.0308 (5)	0.0531 (6)	−0.0127 (4)	−0.0031 (5)	0.0018 (4)
C1	0.0498 (7)	0.0377 (5)	0.0405 (6)	−0.0195 (5)	−0.0069 (5)	0.0020 (5)
C2	0.0699 (9)	0.0460 (6)	0.0484 (7)	−0.0322 (6)	−0.0057 (6)	0.0019 (5)
C3	0.0759 (10)	0.0689 (9)	0.0639 (9)	−0.0489 (8)	−0.0032 (7)	0.0037 (7)
C4	0.0558 (9)	0.0781 (10)	0.0751 (10)	−0.0359 (8)	0.0017 (7)	0.0036 (8)
C5	0.0497 (8)	0.0514 (7)	0.0646 (8)	−0.0187 (6)	0.0018 (6)	0.0002 (6)
C6	0.0473 (7)	0.0388 (6)	0.0441 (6)	−0.0185 (5)	−0.0046 (5)	0.0010 (5)
C7	0.0414 (6)	0.0314 (5)	0.0568 (7)	−0.0124 (5)	−0.0083 (5)	0.0001 (5)
C8	0.0425 (6)	0.0357 (5)	0.0464 (6)	−0.0150 (5)	−0.0063 (5)	0.0031 (5)
C9	0.0455 (6)	0.0333 (5)	0.0435 (6)	−0.0166 (5)	−0.0090 (5)	−0.0019 (4)
C10	0.0425 (6)	0.0368 (5)	0.0556 (7)	−0.0158 (5)	−0.0066 (5)	−0.0018 (5)
C11	0.0469 (7)	0.0394 (6)	0.0688 (9)	−0.0151 (6)	−0.0122 (6)	−0.0043 (6)
C12	0.0465 (8)	0.0482 (7)	0.1026 (12)	−0.0105 (6)	−0.0050 (8)	−0.0099 (8)
C13	0.0605 (8)	0.0414 (6)	0.0596 (8)	−0.0157 (6)	−0.0004 (6)	−0.0132 (6)
C14	0.0638 (9)	0.0600 (8)	0.0493 (7)	−0.0262 (7)	0.0052 (6)	−0.0144 (6)
C15	0.0617 (8)	0.0586 (8)	0.0466 (7)	−0.0262 (7)	−0.0004 (6)	−0.0057 (6)
C16	0.0710 (9)	0.0645 (8)	0.0481 (7)	−0.0306 (8)	−0.0023 (6)	−0.0034 (6)
C17	0.0704 (9)	0.0626 (8)	0.0512 (8)	−0.0294 (8)	−0.0069 (7)	−0.0011 (6)
C18	0.0774 (10)	0.0632 (8)	0.0546 (8)	−0.0309 (8)	−0.0082 (7)	0.0008 (7)
C19	0.0772 (10)	0.0621 (8)	0.0581 (9)	−0.0304 (8)	−0.0100 (7)	−0.0001 (7)
C20	0.0874 (12)	0.0642 (9)	0.0642 (9)	−0.0366 (9)	−0.0170 (8)	0.0040 (7)
C21	0.0953 (13)	0.0717 (10)	0.0833 (12)	−0.0334 (10)	−0.0272 (10)	0.0032 (9)
C22	0.149 (2)	0.0893 (13)	0.1018 (16)	−0.0534 (14)	−0.0625 (14)	0.0264 (12)

Geometric parameters (Å, º)

O1—C7	1.2190 (14)	C13—H13A	0.9700
O2—C11	1.2415 (16)	C13—H13B	0.9700
N1—C7	1.3640 (16)	C14—C15	1.515 (2)
N1—C6	1.4295 (15)	C14—H14A	0.9700
N1—C13	1.4692 (16)	C14—H14B	0.9700
N2—C9	1.3579 (15)	C15—C16	1.5210 (19)
N2—C1	1.4060 (16)	C15—H15A	0.9700
N2—H2A	0.887 (16)	C15—H15B	0.9700
C1—C2	1.3955 (16)	C16—C17	1.512 (2)
C1—C6	1.4007 (16)	C16—H16A	0.9700
C2—C3	1.374 (2)	C16—H16B	0.9700
C2—H2	0.9300	C17—C18	1.525 (2)
C3—C4	1.384 (2)	C17—H17A	0.9700
C3—H3	0.9300	C17—H17B	0.9700
C4—C5	1.377 (2)	C18—C19	1.504 (2)
C4—H4	0.9300	C18—H18A	0.9700
C5—C6	1.3927 (18)	C18—H18B	0.9700
C5—H5	0.9300	C19—C20	1.526 (2)
C7—C8	1.5115 (17)	C19—H19A	0.9700
C8—C9	1.5054 (15)	C19—H19B	0.9700
C8—H8A	0.9700	C20—C21	1.490 (2)
C8—H8B	0.9700	C20—H20A	0.9700
C9—C10	1.3612 (17)	C20—H20B	0.9700
C10—C11	1.4367 (17)	C21—C22	1.514 (3)
C10—H10	0.9300	C21—H21A	0.9700
C11—C12	1.498 (2)	C21—H21B	0.9700
C12—H12A	0.9600	C22—H22A	0.9600
C12—H12B	0.9600	C22—H22B	0.9600
C12—H12C	0.9600	C22—H22C	0.9600
C13—C14	1.5208 (19)		
C7—N1—C6	123.72 (10)	C15—C14—C13	114.50 (11)
C7—N1—C13	118.75 (10)	C15—C14—H14A	108.6
C6—N1—C13	117.46 (10)	C13—C14—H14A	108.6
C9—N2—C1	125.52 (10)	C15—C14—H14B	108.6
C9—N2—H2A	111.0 (10)	C13—C14—H14B	108.6
C1—N2—H2A	120.2 (10)	H14A—C14—H14B	107.6
C2—C1—C6	119.63 (12)	C14—C15—C16	113.14 (12)
C2—C1—N2	118.29 (11)	C14—C15—H15A	109.0
C6—C1—N2	122.07 (10)	C16—C15—H15A	109.0
C3—C2—C1	120.68 (13)	C14—C15—H15B	109.0
C3—C2—H2	119.7	C16—C15—H15B	109.0
C1—C2—H2	119.7	H15A—C15—H15B	107.8
C2—C3—C4	119.84 (13)	C17—C16—C15	115.36 (12)
C2—C3—H3	120.1	C17—C16—H16A	108.4
C4—C3—H3	120.1	C15—C16—H16A	108.4

C5—C4—C3	120.01 (14)	C17—C16—H16B	108.4
C5—C4—H4	120.0	C15—C16—H16B	108.4
C3—C4—H4	120.0	H16A—C16—H16B	107.5
C4—C5—C6	121.13 (13)	C16—C17—C18	112.79 (12)
C4—C5—H5	119.4	C16—C17—H17A	109.0
C6—C5—H5	119.4	C18—C17—H17A	109.0
C5—C6—C1	118.53 (11)	C16—C17—H17B	109.0
C5—C6—N1	119.33 (11)	C18—C17—H17B	109.0
C1—C6—N1	122.03 (11)	H17A—C17—H17B	107.8
O1—C7—N1	123.17 (12)	C19—C18—C17	115.60 (13)
O1—C7—C8	121.50 (12)	C19—C18—H18A	108.4
N1—C7—C8	115.32 (10)	C17—C18—H18A	108.4
C9—C8—C7	109.74 (9)	C19—C18—H18B	108.4
C9—C8—H8A	109.7	C17—C18—H18B	108.4
C7—C8—H8A	109.7	H18A—C18—H18B	107.4
C9—C8—H8B	109.7	C18—C19—C20	113.76 (14)
C7—C8—H8B	109.7	C18—C19—H19A	108.8
H8A—C8—H8B	108.2	C20—C19—H19A	108.8
N2—C9—C10	122.13 (10)	C18—C19—H19B	108.8
N2—C9—C8	115.85 (10)	C20—C19—H19B	108.8
C10—C9—C8	122.02 (10)	H19A—C19—H19B	107.7
C9—C10—C11	123.47 (12)	C21—C20—C19	115.22 (15)
C9—C10—H10	118.3	C21—C20—H20A	108.5
C11—C10—H10	118.3	C19—C20—H20A	108.5
O2—C11—C10	122.16 (12)	C21—C20—H20B	108.5
O2—C11—C12	119.60 (12)	C19—C20—H20B	108.5
C10—C11—C12	118.24 (13)	H20A—C20—H20B	107.5
C11—C12—H12A	109.5	C20—C21—C22	114.29 (19)
C11—C12—H12B	109.5	C20—C21—H21A	108.7
H12A—C12—H12B	109.5	C22—C21—H21A	108.7
C11—C12—H12C	109.5	C20—C21—H21B	108.7
H12A—C12—H12C	109.5	C22—C21—H21B	108.7
H12B—C12—H12C	109.5	H21A—C21—H21B	107.6
N1—C13—C14	112.14 (10)	C21—C22—H22A	109.5
N1—C13—H13A	109.2	C21—C22—H22B	109.5
C14—C13—H13A	109.2	H22A—C22—H22B	109.5
N1—C13—H13B	109.2	C21—C22—H22C	109.5
C14—C13—H13B	109.2	H22A—C22—H22C	109.5
H13A—C13—H13B	107.9	H22B—C22—H22C	109.5

C9—N2—C1—C2	−133.51 (13)	O1—C7—C8—C9	−106.01 (13)
C9—N2—C1—C6	46.96 (17)	N1—C7—C8—C9	72.89 (13)
C6—C1—C2—C3	−1.84 (19)	C1—N2—C9—C10	166.11 (11)
N2—C1—C2—C3	178.62 (12)	C1—N2—C9—C8	−13.82 (17)
C1—C2—C3—C4	−1.9 (2)	C7—C8—C9—N2	−63.37 (13)
C2—C3—C4—C5	3.0 (2)	C7—C8—C9—C10	116.70 (12)
C3—C4—C5—C6	−0.4 (2)	N2—C9—C10—C11	−2.25 (19)
C4—C5—C6—C1	−3.3 (2)	C8—C9—C10—C11	177.68 (11)

C4—C5—C6—N1	172.92 (13)	C9—C10—C11—O2	6.2 (2)
C2—C1—C6—C5	4.37 (18)	C9—C10—C11—C12	−173.27 (12)
N2—C1—C6—C5	−176.11 (11)	C7—N1—C13—C14	115.76 (13)
C2—C1—C6—N1	−171.75 (11)	C6—N1—C13—C14	−61.51 (15)
N2—C1—C6—N1	7.77 (18)	N1—C13—C14—C15	−58.53 (16)
C7—N1—C6—C5	135.27 (13)	C13—C14—C15—C16	−175.86 (12)
C13—N1—C6—C5	−47.61 (15)	C14—C15—C16—C17	−178.65 (12)
C7—N1—C6—C1	−48.64 (16)	C15—C16—C17—C18	−178.22 (13)
C13—N1—C6—C1	128.48 (12)	C16—C17—C18—C19	−178.91 (13)
C6—N1—C7—O1	178.57 (11)	C17—C18—C19—C20	179.79 (13)
C13—N1—C7—O1	1.49 (17)	C18—C19—C20—C21	−179.32 (14)
C6—N1—C7—C8	−0.31 (16)	C19—C20—C21—C22	179.61 (15)
C13—N1—C7—C8	−177.39 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.887 (16)	1.904 (16)	2.6459 (14)	140.0 (14)
C5—H5···O2^{i}	0.93	2.54	3.3123 (17)	140

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