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

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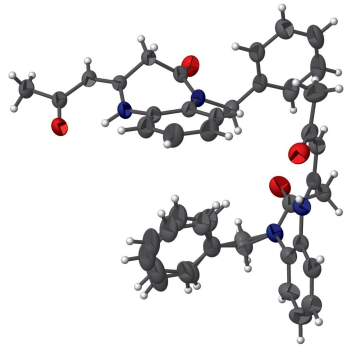
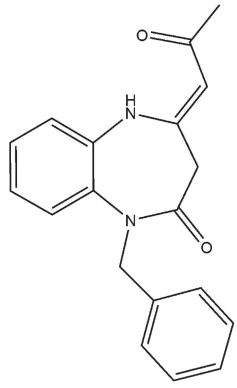
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{19}H_{18}N_2O_2$, crystallizes with two independent molecules in the asymmetric unit which differ in conformation. The seven-membered ring adopts a 'bowl' conformation with the benzyl group oriented away from the open face in one molecule, while the benzyl group is oriented towards the open face in the other. The benzyl group of one independent molecule is disordered over two sets of sites with refined site-occupancy factors of 0.454 (8) and 0.546 (8). The two molecules are linked *via* C—H···O hydrogen bonds and centrosymmetrically related pairs of molecules form dimers through C—H···O hydrogen bonds, packing in rows parallel to the *c* axis.

3D view



Chemical scheme



Structure description

1,5-Benzodiazepinone derivatives display a variety of biological activities (Zellou *et al.*, 1998; Brambilla *et al.*, 2007). As a continuation of our studies of 1,5-benzodiazepinone (Minnih *et al.*, 2014), we have studied the alkylation of (4*Z*)-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1*H*-1,5 benzodiazapin-2-one with benzylchloride.

The asymmetric unit consists of two independent molecules which differ in conformation as indicated by, among others, the C1—N1—C7—C8 torsion angle of −5.6 (2)° in molecule 1 and the corresponding C21–N3–C26–C27 angle of 0.9 (2)° in molecule 2. The orientation of the 2-oxopropylidene substituent is determined by the intramolecular N—H···O hydrogen bond in each molecule (Table 1 and Fig. 1). The seven-membered rings

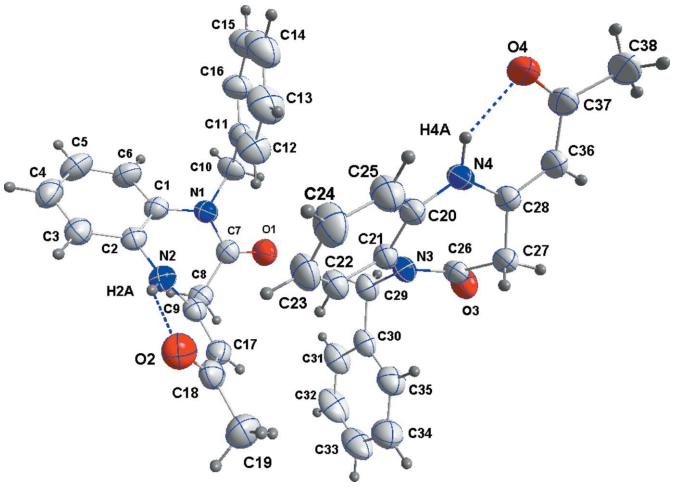


Figure 1
The asymmetric unit with the labeling scheme and 50% probability ellipsoids. Intramolecular hydrogen bonds are shown as dotted lines.

adopt 'bowl' conformations with the benzyl group in molecule 1 oriented away from the open side of the bowl, while that in molecule 2 is oriented towards the open side. Puckering analysis of the seven-membered rings give $Q(2)$ = 0.872 (1), $Q(3)$ = 0.208 (1) Å, $\varphi(2)$ = 206.93 (3) and $\varphi(3)$ = 303.3 (4)° for molecule 1, while for molecule 2 the corresponding values are 0.814 (1), 0.199 (1) Å, 207.66 (9) and 313.3 (4)°. These values also indicate different conformations for the two independent molecules.

In the crystal, pairwise C27—H27*B*···O3^{i} [symmetry code: (i) $-x$, $-y$ + 1, $-z$ + 2] hydrogen bonds between centrosymmetrically related pairs of molecule 2 form weak dimers with each member of the pair making a weak C29—H29*A*···O2^{ii} [symmetry code: (ii) $x-1$, y, z or $1-x$, $1-y$, $2-z$] hydrogen bond to molecule 1 (Table 1, Figs. 2 and 3). These entities then pack in rows extending parallel to the *c* axis.

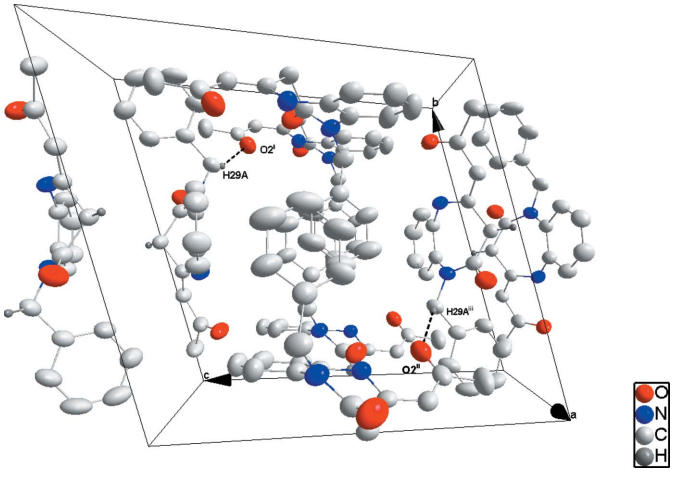


Figure 2
Packing viewed along the *a* axis with C—H···O hydrogen bonds shown as dotted lines [symmetry codes: (i) $-1+x$, y, z; (ii) $2-x$, $1-y$, $1-z$; (iii) $1-x$, $1-y$, $1-z$].

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O2	0.903 (16)	1.906 (16)	2.6242 (14)	135.1 (13)
N4—H4*A*···O4	0.900 (16)	1.914 (16)	2.6417 (14)	136.6 (13)
C27—H27*B*···O3^{i}	0.97	2.48	3.4045 (15)	160
C29—H29*A*···O2^{ii}	0.97	2.48	3.3881 (15)	155

Symmetry codes: (i) $-x$, $-y+1$, $-z+2$; (ii) $x-1$, y, z.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{18}N_2O_2$
M_r	306.35
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	8.2829 (2), 13.8636 (4), 15.1292 (4)
α, β, γ (°)	72.328 (1), 74.922 (1), 87.252 (1)
V (Å^3)	1597.42 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.37 × 0.34 × 0.25
Data collection	
Diffractometer	Bruker *SMART APEX* CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.86, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30572, 8213, 5744
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.151, 1.10
No. of reflections	8213
No. of parameters	422
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.18

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL-2014/7* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 1999) and *SHELXTL* (Sheldrick, 2008).

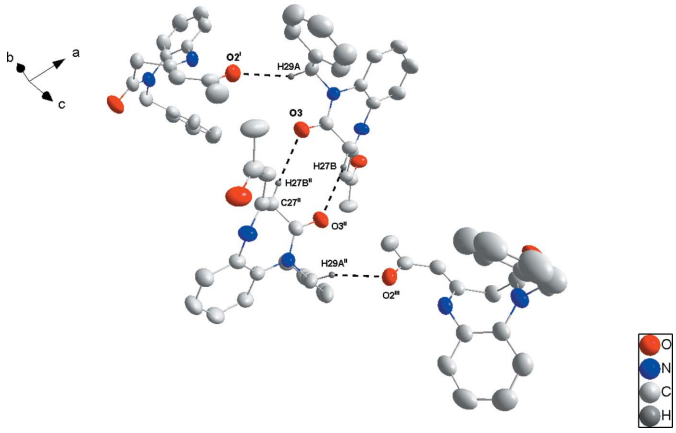


Figure 3
Detail of the C—H···O hydrogen bonding [symmetry codes: (i) $-1+x$, y, z; (ii) $-x$, $1-y$, $1-z$; (iii) $1-x$, $1-y$, $2-z$].

Synthesis and crystallization

To a solution of (4*Z*)-4-(2-oxopropylidene)-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazapin-2-one (500.00 mg) in 10 ml THF were added benzylchloride (263.97 mmol), K_2CO_3 (319.44 mg) and a catalytic amount of tetrabutylammonium bromide. The mixture was stirred at room temperature for 24 h. The solid material was removed by filtration and the solvent evaporated under vacuum. The solid product was purified by recrystallization from ethanol solution to afford crystals in 60% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The benzyl group (C10–C16) of molecule 1 is disordered over two sets of sites with refined site-occupancy factors of 0.454 (8) and 0.546 (8). The disordered groups were refined as rigid hexagons.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{19}H_{18}N_2O_2$
M_r = 306.35
Triclinic, $P\overline{1}$
a = 8.2829 (2) Å
b = 13.8636 (4) Å
c = 15.1292 (4) Å
α = 72.328 (1)°
β = 74.922 (1)°
γ = 87.252 (1)°
V = 1597.42 (7) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.274 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9988 reflections
θ = 2.4–28.6°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.37 × 0.34 × 0.25 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} = 0.86, T_{max} = 0.98
30572 measured reflections
8213 independent reflections
5744 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.7°, θ_{min} = 1.5°
h = −11→11
k = −18→18
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.151
S = 1.10
8213 reflections
422 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2 + 0.0054P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, colllected 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 15 sec/frame.

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). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. The C10–C16 benzyl group is disordered over two sites in approximately equal amounts. The disordered phenyl groups were refined as rigid hexagons.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.49459 (11)	0.85575 (8)	0.49559 (8)	0.0683 (3)	
O2	1.07378 (12)	0.80390 (8)	0.65797 (7)	0.0590 (3)	
N1	0.73037 (12)	0.80871 (8)	0.40458 (8)	0.0466 (3)	
N2	0.98549 (12)	0.83886 (8)	0.49687 (8)	0.0442 (2)	
H2A	1.0618 (19)	0.8162 (12)	0.5309 (11)	0.065 (4)*	
C1	0.90442 (15)	0.82465 (9)	0.35598 (9)	0.0438 (3)	
C2	1.02603 (14)	0.83627 (8)	0.40192 (9)	0.0406 (3)	
C3	1.19430 (16)	0.84348 (10)	0.35267 (10)	0.0509 (3)	
H3	1.2749	0.8502	0.3834	0.061*	
C4	1.24359 (19)	0.84082 (11)	0.25973 (11)	0.0616 (4)	
H4	1.3567	0.8442	0.2282	0.074*	
C5	1.1239 (2)	0.83307 (12)	0.21310 (11)	0.0669 (4)	
H5	1.1563	0.8334	0.1494	0.080*	
C6	0.95696 (18)	0.82488 (11)	0.26089 (10)	0.0590 (4)	
H6	0.8776	0.8194	0.2289	0.071*	
C7	0.64526 (15)	0.86655 (10)	0.45929 (9)	0.0465 (3)	
C8	0.74965 (15)	0.94408 (9)	0.47414 (9)	0.0458 (3)	
H8A	0.8168	0.9852	0.4125	0.055*	
H8B	0.6769	0.9883	0.5044	0.055*	
C9	0.86142 (14)	0.89170 (8)	0.53617 (9)	0.0408 (3)	
C10A	0.6436 (13)	0.7301 (3)	0.3848 (10)	0.0523 (12)	0.546 (8)
H10A	0.6663	0.7436	0.3158	0.063*	0.546 (8)
H10B	0.5239	0.7341	0.4097	0.063*	0.546 (8)
C11A	0.6960 (17)	0.6242 (5)	0.4286 (7)	0.0493 (4)	0.546 (8)
C12A	0.7509 (13)	0.5829 (5)	0.5112 (6)	0.0647 (8)	0.546 (8)
H12A	0.7589	0.6229	0.5494	0.078*	0.546 (8)
C13A	0.7939 (10)	0.4819 (5)	0.5366 (5)	0.0796 (14)	0.546 (8)
H13A	0.8306	0.4543	0.5919	0.096*	0.546 (8)
C14A	0.7819 (10)	0.4221 (4)	0.4795 (6)	0.087 (2)	0.546 (8)

H14A	0.8107	0.3545	0.4965	0.104*	0.546 (8)
C15A	0.7271 (13)	0.4634 (6)	0.3969 (6)	0.0868 (16)	0.546 (8)
H15A	0.7191	0.4234	0.3586	0.104*	0.546 (8)
C16A	0.6841 (17)	0.5644 (6)	0.3714 (7)	0.0687 (8)	0.546 (8)
H16A	0.6474	0.5919	0.3161	0.082*	0.546 (8)
C10B	0.6258 (16)	0.7301 (4)	0.3967 (13)	0.0523 (12)	0.454 (8)
H10C	0.6145	0.7484	0.3314	0.063*	0.454 (8)
H10D	0.5149	0.7289	0.4388	0.063*	0.454 (8)
C11B	0.694 (2)	0.6252 (6)	0.4217 (8)	0.0493 (4)	0.454 (8)
C12B	0.7302 (16)	0.6054 (6)	0.5101 (8)	0.0647 (8)	0.454 (8)
H12B	0.7242	0.6567	0.5386	0.078*	0.454 (8)
C13B	0.7753 (12)	0.5090 (6)	0.5560 (6)	0.0796 (14)	0.454 (8)
H13B	0.7994	0.4958	0.6152	0.096*	0.454 (8)
C14B	0.7843 (12)	0.4324 (5)	0.5134 (7)	0.087 (2)	0.454 (8)
H14B	0.8144	0.3678	0.5441	0.104*	0.454 (8)
C15B	0.7482 (16)	0.4521 (7)	0.4250 (8)	0.0868 (16)	0.454 (8)
H15B	0.7542	0.4009	0.3965	0.104*	0.454 (8)
C16B	0.703 (2)	0.5486 (8)	0.3791 (8)	0.0687 (8)	0.454 (8)
H16B	0.6790	0.5618	0.3199	0.082*	0.454 (8)
C17	0.84035 (16)	0.89753 (9)	0.62720 (9)	0.0468 (3)	
H17	0.7499	0.9323	0.6522	0.056*	
C18	0.95154 (17)	0.85246 (10)	0.68563 (9)	0.0490 (3)	
C19	0.9189 (2)	0.86724 (14)	0.78271 (11)	0.0737 (5)	
H19A	0.8048	0.8855	0.8023	0.111*	
H19B	0.9923	0.9203	0.7798	0.111*	
H19C	0.9387	0.8055	0.8282	0.111*	
O3	0.12636 (11)	0.61188 (7)	0.89024 (7)	0.0588 (3)	
O4	0.34410 (13)	0.20073 (7)	0.86910 (7)	0.0589 (3)	
N3	0.40017 (12)	0.60368 (7)	0.82003 (7)	0.0406 (2)	
N4	0.44056 (13)	0.38238 (8)	0.86210 (8)	0.0445 (2)	
H4A	0.4635 (18)	0.3241 (12)	0.8475 (11)	0.063 (4)*	
C20	0.57574 (14)	0.45236 (8)	0.83664 (9)	0.0407 (3)	
C21	0.55810 (14)	0.55738 (8)	0.81564 (8)	0.0380 (2)	
C22	0.70389 (15)	0.61834 (9)	0.78480 (9)	0.0465 (3)	
H22	0.6949	0.6882	0.7705	0.056*	
C23	0.86031 (16)	0.57807 (11)	0.77493 (11)	0.0568 (3)	
H23	0.9551	0.6206	0.7531	0.068*	
C24	0.87611 (17)	0.47499 (12)	0.79733 (12)	0.0642 (4)	
H24	0.9814	0.4472	0.7920	0.077*	
C25	0.73507 (17)	0.41329 (10)	0.82774 (11)	0.0572 (4)	
H25	0.7463	0.3435	0.8428	0.069*	
C26	0.25564 (14)	0.56650 (9)	0.88922 (9)	0.0405 (3)	
C27	0.26317 (15)	0.46805 (8)	0.96621 (8)	0.0412 (3)	
H27A	0.3517	0.4738	0.9957	0.049*	
H27B	0.1583	0.4560	1.0156	0.049*	
C28	0.29436 (14)	0.37989 (8)	0.92724 (8)	0.0382 (2)	
C29	0.39259 (16)	0.70696 (9)	0.75626 (9)	0.0466 (3)	
H29A	0.2886	0.7128	0.7372	0.056*	

H29B	0.4838	0.7178	0.6987	0.056*
C30	0.40341 (14)	0.78903 (9)	0.80187 (9)	0.0441 (3)
C31	0.38845 (18)	0.88874 (10)	0.74948 (12)	0.0617 (4)
H31	0.3729	0.9033	0.6881	0.074*
C32	0.3964 (2)	0.96637 (11)	0.78740 (16)	0.0775 (5)
H32	0.3859	1.0328	0.7514	0.093*
C33	0.4194 (2)	0.94693 (12)	0.87772 (15)	0.0735 (5)
H33	0.4242	0.9998	0.9030	0.088*
C34	0.4355 (2)	0.84830 (12)	0.93071 (12)	0.0656 (4)
H34	0.4522	0.8343	0.9917	0.079*
C35	0.42654 (18)	0.77051 (10)	0.89266 (10)	0.0558 (3)
H35	0.4363	0.7041	0.9290	0.067*
C36	0.18214 (15)	0.29997 (9)	0.95846 (9)	0.0438 (3)
H36	0.0788	0.3046	0.9995	0.053*
C37	0.21612 (17)	0.20983 (9)	0.93104 (9)	0.0470 (3)
C38	0.0953 (2)	0.12070 (11)	0.98263 (11)	0.0659 (4)
H38A	−0.0136	0.1443	1.0058	0.099*
H38B	0.1326	0.0773	1.0358	0.099*
H38C	0.0899	0.0836	0.9392	0.099*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0388 (5)	0.0776 (7)	0.0923 (8)	0.0071 (5)	−0.0101 (5)	−0.0376 (6)
O2	0.0570 (6)	0.0631 (6)	0.0557 (6)	0.0055 (5)	−0.0226 (5)	−0.0102 (5)
N1	0.0393 (5)	0.0448 (6)	0.0609 (7)	0.0066 (4)	−0.0170 (5)	−0.0208 (5)
N2	0.0415 (5)	0.0448 (6)	0.0493 (6)	0.0121 (4)	−0.0173 (5)	−0.0154 (5)
C1	0.0426 (6)	0.0407 (6)	0.0499 (7)	0.0085 (5)	−0.0136 (5)	−0.0158 (5)
C2	0.0410 (6)	0.0342 (6)	0.0467 (7)	0.0082 (5)	−0.0122 (5)	−0.0126 (5)
C3	0.0420 (7)	0.0456 (7)	0.0653 (9)	0.0077 (5)	−0.0123 (6)	−0.0195 (6)
C4	0.0501 (8)	0.0577 (8)	0.0698 (10)	0.0104 (7)	0.0002 (7)	−0.0236 (7)
C5	0.0731 (10)	0.0743 (10)	0.0538 (8)	0.0177 (8)	−0.0081 (7)	−0.0291 (8)
C6	0.0611 (9)	0.0685 (9)	0.0573 (8)	0.0144 (7)	−0.0212 (7)	−0.0300 (7)
C7	0.0399 (6)	0.0456 (7)	0.0551 (7)	0.0098 (5)	−0.0166 (6)	−0.0144 (6)
C8	0.0447 (6)	0.0379 (6)	0.0563 (7)	0.0120 (5)	−0.0156 (6)	−0.0158 (5)
C9	0.0394 (6)	0.0329 (6)	0.0492 (7)	0.0027 (5)	−0.0114 (5)	−0.0115 (5)
C10A	0.0464 (19)	0.0526 (8)	0.067 (3)	0.0040 (7)	−0.025 (2)	−0.0225 (8)
C11A	0.0382 (7)	0.0492 (7)	0.0585 (13)	−0.0008 (5)	−0.0085 (9)	−0.0163 (7)
C12A	0.068 (2)	0.047 (2)	0.0763 (11)	0.017 (2)	−0.0300 (12)	−0.0073 (19)
C13A	0.084 (2)	0.046 (3)	0.099 (3)	0.013 (2)	−0.029 (2)	−0.0057 (19)
C14A	0.0731 (13)	0.0451 (15)	0.131 (6)	0.0032 (13)	−0.021 (3)	−0.016 (2)
C15A	0.081 (3)	0.0571 (18)	0.130 (5)	0.0077 (15)	−0.031 (3)	−0.039 (3)
C16A	0.068 (3)	0.052 (2)	0.0905 (17)	0.0066 (17)	−0.0168 (11)	−0.0321 (17)
C10B	0.0464 (19)	0.0526 (8)	0.067 (3)	0.0040 (7)	−0.025 (2)	−0.0225 (8)
C11B	0.0382 (7)	0.0492 (7)	0.0585 (13)	−0.0008 (5)	−0.0085 (9)	−0.0163 (7)
C12B	0.068 (2)	0.047 (2)	0.0763 (11)	0.017 (2)	−0.0300 (12)	−0.0073 (19)
C13B	0.084 (2)	0.046 (3)	0.099 (3)	0.013 (2)	−0.029 (2)	−0.0057 (19)
C14B	0.0731 (13)	0.0451 (15)	0.131 (6)	0.0032 (13)	−0.021 (3)	−0.016 (2)

C15B	0.081 (3)	0.0571 (18)	0.130 (5)	0.0077 (15)	−0.031 (3)	−0.039 (3)
C16B	0.068 (3)	0.052 (2)	0.0905 (17)	0.0066 (17)	−0.0168 (11)	−0.0321 (17)
C17	0.0452 (7)	0.0433 (7)	0.0506 (7)	−0.0005 (5)	−0.0078 (5)	−0.0160 (5)
C18	0.0500 (7)	0.0472 (7)	0.0446 (7)	−0.0122 (6)	−0.0084 (6)	−0.0074 (5)
C19	0.0802 (11)	0.0906 (12)	0.0507 (9)	−0.0111 (9)	−0.0170 (8)	−0.0196 (8)
O3	0.0403 (5)	0.0525 (5)	0.0753 (7)	0.0070 (4)	−0.0112 (4)	−0.0113 (5)
O4	0.0678 (6)	0.0465 (5)	0.0602 (6)	−0.0094 (5)	−0.0012 (5)	−0.0241 (4)
N3	0.0389 (5)	0.0337 (5)	0.0462 (6)	0.0006 (4)	−0.0122 (4)	−0.0067 (4)
N4	0.0431 (5)	0.0354 (5)	0.0525 (6)	−0.0042 (4)	−0.0022 (5)	−0.0175 (5)
C20	0.0383 (6)	0.0363 (6)	0.0452 (6)	−0.0025 (5)	−0.0055 (5)	−0.0126 (5)
C21	0.0371 (6)	0.0361 (6)	0.0390 (6)	−0.0006 (5)	−0.0080 (5)	−0.0099 (5)
C22	0.0443 (6)	0.0387 (6)	0.0525 (7)	−0.0058 (5)	−0.0095 (5)	−0.0097 (5)
C23	0.0382 (6)	0.0560 (8)	0.0691 (9)	−0.0084 (6)	−0.0059 (6)	−0.0142 (7)
C24	0.0357 (6)	0.0600 (9)	0.0865 (11)	0.0059 (6)	−0.0055 (7)	−0.0163 (8)
C25	0.0469 (7)	0.0418 (7)	0.0741 (10)	0.0052 (6)	−0.0042 (7)	−0.0152 (6)
C26	0.0369 (6)	0.0369 (6)	0.0497 (7)	0.0001 (5)	−0.0110 (5)	−0.0158 (5)
C27	0.0430 (6)	0.0371 (6)	0.0418 (6)	−0.0026 (5)	−0.0056 (5)	−0.0132 (5)
C28	0.0408 (6)	0.0345 (6)	0.0371 (6)	0.0002 (5)	−0.0086 (5)	−0.0087 (5)
C29	0.0488 (7)	0.0390 (6)	0.0468 (7)	0.0022 (5)	−0.0162 (6)	−0.0025 (5)
C30	0.0363 (6)	0.0354 (6)	0.0549 (7)	0.0008 (5)	−0.0115 (5)	−0.0054 (5)
C31	0.0588 (8)	0.0430 (7)	0.0760 (10)	0.0037 (6)	−0.0259 (7)	−0.0005 (7)
C32	0.0767 (11)	0.0352 (7)	0.1137 (15)	0.0069 (7)	−0.0295 (10)	−0.0091 (8)
C33	0.0659 (10)	0.0487 (8)	0.1082 (14)	0.0008 (7)	−0.0135 (9)	−0.0345 (9)
C34	0.0710 (10)	0.0572 (9)	0.0718 (10)	0.0001 (7)	−0.0160 (8)	−0.0260 (8)
C35	0.0664 (9)	0.0392 (7)	0.0595 (8)	0.0015 (6)	−0.0186 (7)	−0.0097 (6)
C36	0.0431 (6)	0.0396 (6)	0.0446 (7)	−0.0062 (5)	−0.0062 (5)	−0.0101 (5)
C37	0.0553 (7)	0.0415 (6)	0.0452 (7)	−0.0081 (6)	−0.0132 (6)	−0.0129 (5)
C38	0.0844 (11)	0.0510 (8)	0.0583 (9)	−0.0267 (8)	−0.0064 (8)	−0.0164 (7)

Geometric parameters (Å, º)

O1—C7	1.2218 (15)	C16B—H16B	0.9300
O2—C18	1.2405 (16)	C17—C18	1.4328 (18)
N1—C7	1.3676 (16)	C17—H17	0.9300
N1—C1	1.4311 (16)	C18—C19	1.498 (2)
N1—C10B	1.475 (3)	C19—H19A	0.9600
N1—C10A	1.476 (3)	C19—H19B	0.9600
N2—C9	1.3493 (14)	C19—H19C	0.9600
N2—C2	1.3989 (16)	O3—C26	1.2147 (13)
N2—H2A	0.903 (16)	O4—C37	1.2489 (15)
C1—C6	1.3895 (19)	N3—C26	1.3690 (15)
C1—C2	1.4041 (16)	N3—C21	1.4240 (14)
C2—C3	1.3912 (17)	N3—C29	1.4728 (14)
C3—C4	1.370 (2)	N4—C28	1.3421 (15)
C3—H3	0.9300	N4—C20	1.4129 (15)
C4—C5	1.383 (2)	N4—H4A	0.900 (16)
C4—H4	0.9300	C20—C25	1.3916 (17)
C5—C6	1.376 (2)	C20—C21	1.4018 (16)

C5—H5	0.9300	C21—C22	1.3983 (16)
C6—H6	0.9300	C22—C23	1.3759 (18)
C7—C8	1.5134 (17)	C22—H22	0.9300
C8—C9	1.4962 (16)	C23—C24	1.373 (2)
C8—H8A	0.9700	C23—H23	0.9300
C8—H8B	0.9700	C24—C25	1.373 (2)
C9—C17	1.3693 (17)	C24—H24	0.9300
C10A—C11A	1.511 (3)	C25—H25	0.9300
C10A—H10A	0.9700	C26—C27	1.5126 (16)
C10A—H10B	0.9700	C27—C28	1.4954 (15)
C11A—C12A	1.3900	C27—H27A	0.9700
C11A—C16A	1.3900	C27—H27B	0.9700
C12A—C13A	1.3900	C28—C36	1.3665 (16)
C12A—H12A	0.9300	C29—C30	1.5157 (18)
C13A—C14A	1.3900	C29—H29A	0.9700
C13A—H13A	0.9300	C29—H29B	0.9700
C14A—C15A	1.3900	C30—C35	1.3810 (19)
C14A—H14A	0.9300	C30—C31	1.3870 (17)
C15A—C16A	1.3900	C31—C32	1.376 (2)
C15A—H15A	0.9300	C31—H31	0.9300
C16A—H16A	0.9300	C32—C33	1.372 (3)
C10B—C11B	1.509 (4)	C32—H32	0.9300
C10B—H10C	0.9700	C33—C34	1.379 (2)
C10B—H10D	0.9700	C33—H33	0.9300
C11B—C12B	1.3900	C34—C35	1.381 (2)
C11B—C16B	1.3900	C34—H34	0.9300
C12B—C13B	1.3900	C35—H35	0.9300
C12B—H12B	0.9300	C36—C37	1.4259 (17)
C13B—C14B	1.3900	C36—H36	0.9300
C13B—H13B	0.9300	C37—C38	1.5046 (19)
C14B—C15B	1.3900	C38—H38A	0.9600
C14B—H14B	0.9300	C38—H38B	0.9600
C15B—C16B	1.3900	C38—H38C	0.9600
C15B—H15B	0.9300		

C7—N1—C1	123.42 (10)	C11B—C16B—H16B	120.0
C7—N1—C10B	114.2 (7)	C9—C17—C18	122.89 (12)
C1—N1—C10B	122.4 (7)	C9—C17—H17	118.6
C7—N1—C10A	121.4 (5)	C18—C17—H17	118.6
C1—N1—C10A	115.0 (6)	O2—C18—C17	122.48 (12)
C9—N2—C2	125.71 (10)	O2—C18—C19	119.59 (13)
C9—N2—H2A	115.1 (10)	C17—C18—C19	117.92 (13)
C2—N2—H2A	117.5 (10)	C18—C19—H19A	109.5
C6—C1—C2	118.32 (12)	C18—C19—H19B	109.5
C6—C1—N1	119.22 (11)	H19A—C19—H19B	109.5
C2—C1—N1	122.42 (11)	C18—C19—H19C	109.5
C3—C2—N2	117.97 (11)	H19A—C19—H19C	109.5
C3—C2—C1	119.41 (11)	H19B—C19—H19C	109.5

N2—C2—C1	122.61 (11)	C26—N3—C21	124.95 (9)
C4—C3—C2	121.27 (13)	C26—N3—C29	115.38 (9)
C4—C3—H3	119.4	C21—N3—C29	118.90 (10)
C2—C3—H3	119.4	C28—N4—C20	126.37 (10)
C3—C4—C5	119.48 (14)	C28—N4—H4A	114.2 (10)
C3—C4—H4	120.3	C20—N4—H4A	116.9 (10)
C5—C4—H4	120.3	C25—C20—C21	119.28 (11)
C6—C5—C4	120.08 (14)	C25—C20—N4	116.87 (10)
C6—C5—H5	120.0	C21—C20—N4	123.77 (10)
C4—C5—H5	120.0	C22—C21—C20	117.68 (10)
C5—C6—C1	121.38 (13)	C22—C21—N3	119.43 (10)
C5—C6—H6	119.3	C20—C21—N3	122.82 (10)
C1—C6—H6	119.3	C23—C22—C21	121.99 (12)
O1—C7—N1	122.51 (12)	C23—C22—H22	119.0
O1—C7—C8	121.55 (12)	C21—C22—H22	119.0
N1—C7—C8	115.93 (11)	C24—C23—C22	119.87 (12)
C9—C8—C7	109.88 (10)	C24—C23—H23	120.1
C9—C8—H8A	109.7	C22—C23—H23	120.1
C7—C8—H8A	109.7	C23—C24—C25	119.39 (12)
C9—C8—H8B	109.7	C23—C24—H24	120.3
C7—C8—H8B	109.7	C25—C24—H24	120.3
H8A—C8—H8B	108.2	C24—C25—C20	121.76 (12)
N2—C9—C17	121.77 (11)	C24—C25—H25	119.1
N2—C9—C8	116.07 (11)	C20—C25—H25	119.1
C17—C9—C8	122.15 (11)	O3—C26—N3	121.36 (11)
N1—C10A—C11A	113.1 (5)	O3—C26—C27	120.90 (11)
N1—C10A—H10A	109.0	N3—C26—C27	117.72 (10)
C11A—C10A—H10A	109.0	C28—C27—C26	112.29 (10)
N1—C10A—H10B	109.0	C28—C27—H27A	109.1
C11A—C10A—H10B	109.0	C26—C27—H27A	109.1
H10A—C10A—H10B	107.8	C28—C27—H27B	109.1
C12A—C11A—C16A	120.0	C26—C27—H27B	109.1
C12A—C11A—C10A	130.7 (6)	H27A—C27—H27B	107.9
C16A—C11A—C10A	109.3 (6)	N4—C28—C36	121.99 (11)
C13A—C12A—C11A	120.0	N4—C28—C27	116.28 (10)
C13A—C12A—H12A	120.0	C36—C28—C27	121.70 (11)
C11A—C12A—H12A	120.0	N3—C29—C30	113.59 (10)
C12A—C13A—C14A	120.0	N3—C29—H29A	108.8
C12A—C13A—H13A	120.0	C30—C29—H29A	108.8
C14A—C13A—H13A	120.0	N3—C29—H29B	108.8
C15A—C14A—C13A	120.0	C30—C29—H29B	108.8
C15A—C14A—H14A	120.0	H29A—C29—H29B	107.7
C13A—C14A—H14A	120.0	C35—C30—C31	117.99 (13)
C14A—C15A—C16A	120.0	C35—C30—C29	123.96 (11)
C14A—C15A—H15A	120.0	C31—C30—C29	118.06 (12)
C16A—C15A—H15A	120.0	C32—C31—C30	120.64 (15)
C15A—C16A—C11A	120.0	C32—C31—H31	119.7
C15A—C16A—H16A	120.0	C30—C31—H31	119.7

C11A—C16A—H16A	120.0	C33—C32—C31	120.80 (14)
N1—C10B—C11B	113.9 (7)	C33—C32—H32	119.6
N1—C10B—H10C	108.8	C31—C32—H32	119.6
C11B—C10B—H10C	108.8	C32—C33—C34	119.40 (15)
N1—C10B—H10D	108.8	C32—C33—H33	120.3
C11B—C10B—H10D	108.8	C34—C33—H33	120.3
H10C—C10B—H10D	107.7	C33—C34—C35	119.66 (16)
C12B—C11B—C16B	120.0	C33—C34—H34	120.2
C12B—C11B—C10B	108.1 (8)	C35—C34—H34	120.2
C16B—C11B—C10B	131.3 (8)	C34—C35—C30	121.52 (13)
C13B—C12B—C11B	120.0	C34—C35—H35	119.2
C13B—C12B—H12B	120.0	C30—C35—H35	119.2
C11B—C12B—H12B	120.0	C28—C36—C37	123.06 (12)
C12B—C13B—C14B	120.0	C28—C36—H36	118.5
C12B—C13B—H13B	120.0	C37—C36—H36	118.5
C14B—C13B—H13B	120.0	O4—C37—C36	122.92 (11)
C13B—C14B—C15B	120.0	O4—C37—C38	119.04 (12)
C13B—C14B—H14B	120.0	C36—C37—C38	117.98 (12)
C15B—C14B—H14B	120.0	C37—C38—H38A	109.5
C16B—C15B—C14B	120.0	C37—C38—H38B	109.5
C16B—C15B—H15B	120.0	H38A—C38—H38B	109.5
C14B—C15B—H15B	120.0	C37—C38—H38C	109.5
C15B—C16B—C11B	120.0	H38A—C38—H38C	109.5
C15B—C16B—H16B	120.0	H38B—C38—H38C	109.5

C7—N1—C1—C6	−133.87 (13)	C13B—C14B—C15B—C16B	0.0
C10B—N1—C1—C6	43.8 (5)	C14B—C15B—C16B—C11B	0.0
C10A—N1—C1—C6	41.0 (4)	C12B—C11B—C16B—C15B	0.0
C7—N1—C1—C2	48.67 (17)	C10B—C11B—C16B—C15B	−169.7 (16)
C10B—N1—C1—C2	−133.7 (5)	N2—C9—C17—C18	2.93 (19)
C10A—N1—C1—C2	−136.5 (4)	C8—C9—C17—C18	−176.51 (11)
C9—N2—C2—C3	136.54 (12)	C9—C17—C18—O2	−0.9 (2)
C9—N2—C2—C1	−44.82 (17)	C9—C17—C18—C19	177.81 (13)
C6—C1—C2—C3	−2.56 (18)	C28—N4—C20—C25	135.83 (13)
N1—C1—C2—C3	174.93 (11)	C28—N4—C20—C21	−47.47 (18)
C6—C1—C2—N2	178.82 (11)	C25—C20—C21—C22	1.11 (18)
N1—C1—C2—N2	−3.70 (17)	N4—C20—C21—C22	−175.51 (11)
N2—C2—C3—C4	179.57 (12)	C25—C20—C21—N3	177.99 (12)
C1—C2—C3—C4	0.88 (19)	N4—C20—C21—N3	1.38 (18)
C2—C3—C4—C5	1.4 (2)	C26—N3—C21—C22	−143.15 (12)
C3—C4—C5—C6	−2.0 (2)	C29—N3—C21—C22	26.29 (16)
C4—C5—C6—C1	0.3 (2)	C26—N3—C21—C20	40.02 (17)
C2—C1—C6—C5	2.0 (2)	C29—N3—C21—C20	−150.54 (11)
N1—C1—C6—C5	−175.58 (13)	C20—C21—C22—C23	0.02 (19)
C1—N1—C7—O1	175.53 (12)	N3—C21—C22—C23	−176.98 (12)
C10B—N1—C7—O1	−2.3 (5)	C21—C22—C23—C24	−1.2 (2)
C10A—N1—C7—O1	1.0 (5)	C22—C23—C24—C25	1.2 (2)
C1—N1—C7—C8	−5.63 (16)	C23—C24—C25—C20	0.0 (2)

C10B—N1—C7—C8	176.6 (5)	C21—C20—C25—C24	−1.1 (2)
C10A—N1—C7—C8	179.8 (4)	N4—C20—C25—C24	175.73 (13)
O1—C7—C8—C9	110.17 (13)	C21—N3—C26—O3	177.53 (11)
N1—C7—C8—C9	−68.68 (14)	C29—N3—C26—O3	7.76 (16)
C2—N2—C9—C17	−173.75 (12)	C21—N3—C26—C27	−0.89 (16)
C2—N2—C9—C8	5.73 (17)	C29—N3—C26—C27	−170.66 (10)
C7—C8—C9—N2	68.48 (14)	O3—C26—C27—C28	113.86 (13)
C7—C8—C9—C17	−112.04 (13)	N3—C26—C27—C28	−67.71 (13)
C7—N1—C10A—C11A	−114.9 (9)	C20—N4—C28—C36	−169.03 (11)
C1—N1—C10A—C11A	70.1 (12)	C20—N4—C28—C27	9.10 (17)
N1—C10A—C11A—C12A	30.3 (17)	C26—C27—C28—N4	62.87 (13)
N1—C10A—C11A—C16A	−149.3 (8)	C26—C27—C28—C36	−118.99 (12)
C16A—C11A—C12A—C13A	0.0	C26—N3—C29—C30	77.22 (13)
C10A—C11A—C12A—C13A	−179.5 (13)	C21—N3—C29—C30	−93.21 (13)
C11A—C12A—C13A—C14A	0.0	N3—C29—C30—C35	3.06 (18)
C12A—C13A—C14A—C15A	0.0	N3—C29—C30—C31	−176.64 (11)
C13A—C14A—C15A—C16A	0.0	C35—C30—C31—C32	−0.1 (2)
C14A—C15A—C16A—C11A	0.0	C29—C30—C31—C32	179.64 (14)
C12A—C11A—C16A—C15A	0.0	C30—C31—C32—C33	0.1 (3)
C10A—C11A—C16A—C15A	179.6 (11)	C31—C32—C33—C34	0.2 (3)
C7—N1—C10B—C11B	−128.0 (11)	C32—C33—C34—C35	−0.6 (2)
C1—N1—C10B—C11B	54.2 (15)	C33—C34—C35—C30	0.6 (2)
N1—C10B—C11B—C12B	50.8 (16)	C31—C30—C35—C34	−0.3 (2)
N1—C10B—C11B—C16B	−138.6 (10)	C29—C30—C35—C34	180.00 (13)
C16B—C11B—C12B—C13B	0.0	N4—C28—C36—C37	5.37 (18)
C10B—C11B—C12B—C13B	171.9 (12)	C27—C28—C36—C37	−172.66 (11)
C11B—C12B—C13B—C14B	0.0	C28—C36—C37—O4	−7.3 (2)
C12B—C13B—C14B—C15B	0.0	C28—C36—C37—C38	169.96 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.903 (16)	1.906 (16)	2.6242 (14)	135.1 (13)
N4—H4*A*···O4	0.900 (16)	1.914 (16)	2.6417 (14)	136.6 (13)
C27—H27*B*···O3^{i}	0.97	2.48	3.4045 (15)	160
C29—H29*A*···O2^{ii}	0.97	2.48	3.3881 (15)	155

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2; (ii) *x*−1, *y*, *z*.