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2-(2-Oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetic acid

Abdelhanine Essaghouani,[a]* Mohammed Boulhaoua,[a] Mohamed El Hafi,[a] Mohammed Benchidmi,[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, Université Mohammed V, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: essaghouani.hanine@gmail.com

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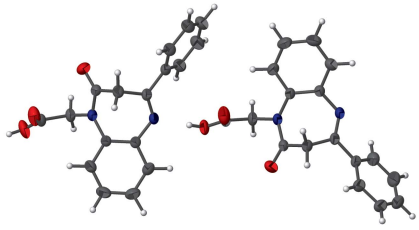
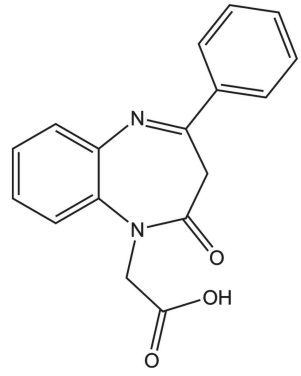
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_{17}H_{14}N_2O_3$, consists of two independent molecules having distinctly different conformations. The components of the asymmetric unit are connected by an O—H···N hydrogen bond, with additional O—H···N hydrogen bonds connecting this assemblage into chains running parallel to the *b* axis. Intermolecular C—H···π(ring) interactions are also present.

3D view



Chemical scheme



Structure description

As a continuation of our studies on substituted 1,5-benzodiazepin-2-one derivatives (Essaghouani *et al.*, 2016; Ballo *et al.*, 2010), we report the synthesis of a new 1,5-benzodiazepin-2-one derivative by the hydrolysis reaction with an aqueous solution of potassium hydroxide of ethyl 2-(2-oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetate in ethanol.

The asymmetric unit (Fig. 1) consists of two independent molecules which differ markedly in their conformations. This can be seen, in part, from the puckering parameters for the seven-membered rings. For the ring N1,C1,C6,N2,C7,C8,C9, $Q(2)$ = 0.881 (1) Å, $Q(3)$ = 0.218 (1) Å, $\varphi(2)$ = 203.79 (7)° and $\varphi(3)$ = 307.7 (3)° with a total puckering amplitude of 0.907 (1) Å, while for the ring N3,C18,C23,N4,C24,C25,C26, the corresponding values are 0.904 (1) Å, 0.234 (1) Å, 25.64 (7)° and 125.9 (3)° with a total puckering amplitude of 0.934 (1) Å. Additionally, the dihedral angle between the C1–C6 and C12–C17 rings is 72.18 (4)° while that between the C18–C22 and C29–C34 rings is 80.03 (4)°.

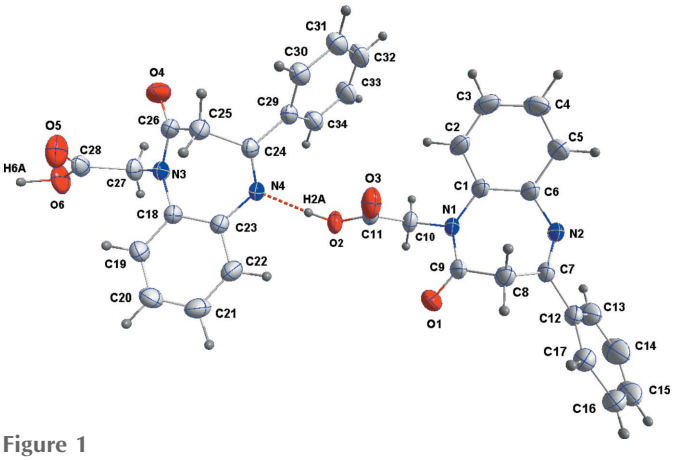


Figure 1
The asymmetric unit with the atom-labelling scheme and 50% probability ellipsoids. The O—H···N hydrogen bond is shown by the dotted line.

In the crystal, the molecules are linked into chains running parallel to the *b* axis by O2—H2*A*···N4 and O6—H6···N2^{iii} [symmetry code: (iii) $x, y+1, z$] hydrogen bonds (Table 1 and Fig. 2). Finally there are two sets of C—H···π(ring) interactions (Fig. 3).

Synthesis and crystallization

A solution of potassium hydroxide (12.0 mmol) in water (5 ml) was added to the solution of ethyl 2-(2-oxo-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepin-1-yl)acetate (3.0 mmol) in ethanol (10 ml). The resulting reaction mixture was stirred at room temperature for 6 h, after completion of the reaction (monitored by TLC). The reaction mixture was poured into water and acidified with 3*M* HCl to form the title compound as a yellow solid. The solid product was purified by recrystallization from ethanol solution to afford the title compound as yellow crystals (yield 84%).

Refinement

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

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

*Cg*1 and *Cg*2 are the centroids of the C18–C22 and C29–C34 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N4	0.82	1.90	2.7161 (12)	176
O6—H6···N2^{i}	0.82	1.95	2.7585 (13)	171
C4—H4···*Cg*2^{ii}	0.989 (16)	2.97 (2)	3.7650 (16)	139 (1)
C17—H17···*Cg*1^{iii}	1.004 (17)	2.82 (2)	3.6431 (14)	140 (1)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{14}N_2O_3$
M_r	294.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, *b*, *c* (Å)	17.9752 (8), 17.4403 (8), 9.2472 (4)
β (°)	102.892 (1)
V (Å^3)	2825.9 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.32 × 0.29 × 0.28
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.89, 0.97
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	54166, 7627, 5791
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.687
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.048, 0.146, 1.11
No. of reflections	7627
No. of parameters	503
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.21

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

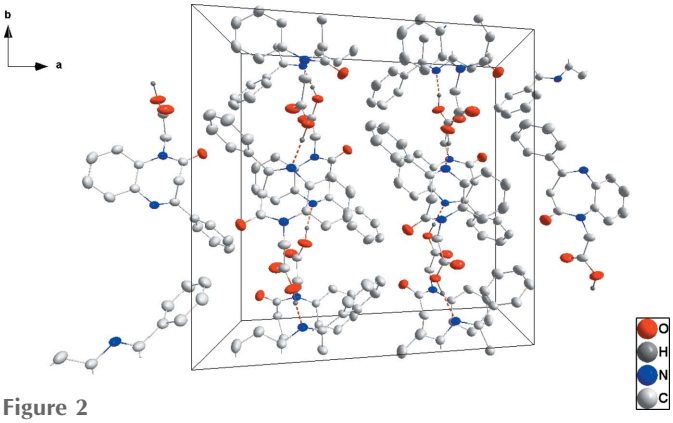


Figure 2
Packing viewed along the *c* axis, with O—H···N hydrogen bonds shown as dotted lines.

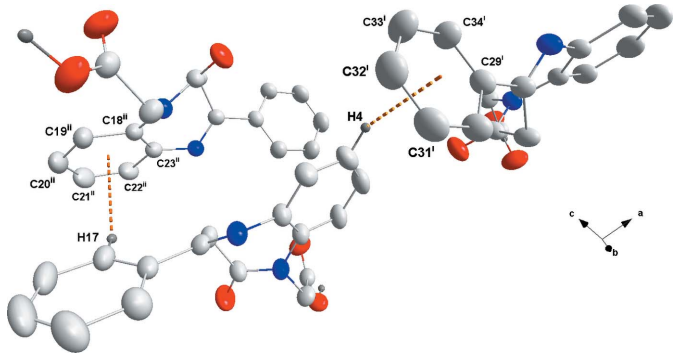


Figure 3
Detail of the C—H···π(ring) interactions (dotted lines) [symmetry codes: (i) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$].

Acknowledgements

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References

Ballo, D., Ahabchane, N. H., Zouihri, H., Essassi, E. M. & Ng, S. W. (2010). *Acta Cryst.* E**66**, o2070.
Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2016). *APEX3*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Essaghouani, A., Bouzian, Y., Essassi, E. M., Saadi, M. & El Ammari, L. (2016). *IUCrData*, **1**, x160661.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2017). **2**, x170120 [https://doi.org/10.1107/S2414314617001201]

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Crystal data

$C_{17}H_{14}N_2O_3$
M_r = 294.30
Monoclinic, $P2_1/c$
a = 17.9752 (8) Å
b = 17.4403 (8) Å
c = 9.2472 (4) Å
β = 102.892 (1)°
V = 2825.9 (2) Å^3
Z = 8
$F(000)$ = 1232
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9857 reflections
θ = 2.3–28.8°
μ = 0.10 mm^{-1}
T = 298 K
Block, yellow
0.32 × 0.29 × 0.28 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} = 0.89, T_{max} = 0.97
54166 measured reflections
7627 independent reflections
5791 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 29.2°, θ_{min} = 1.7°
h = −24→24
k = −23→23
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.146
S = 1.11
7627 reflections
503 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
$w = 1/[\sigma^2(F_o^2) + (0.0941P)^2 + 0.0156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 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. Hydrogens attached to oxygen were placed in calculated positions and included as riding contributions.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.60331 (5)	0.13791 (5)	0.27570 (13)	0.0536 (3)
O2	0.70218 (5)	0.30973 (5)	0.14443 (10)	0.0448 (2)
H2A	0.7194	0.3483	0.1907	0.067*
O3	0.76238 (7)	0.24959 (5)	0.34987 (10)	0.0523 (3)
N1	0.71781 (5)	0.10783 (5)	0.22680 (11)	0.0335 (2)
N2	0.72769 (5)	−0.06012 (5)	0.28503 (11)	0.0338 (2)
C1	0.78810 (6)	0.06741 (6)	0.26215 (12)	0.0325 (2)
C2	0.85619 (7)	0.10630 (7)	0.26274 (15)	0.0429 (3)
H2	0.8576 (8)	0.1607 (10)	0.2436 (17)	0.054 (4)*
C3	0.92572 (8)	0.06889 (9)	0.29592 (17)	0.0498 (3)
H3	0.9738 (9)	0.0964 (10)	0.3011 (19)	0.061 (4)*
C4	0.92926 (7)	−0.00864 (9)	0.32758 (16)	0.0488 (3)
H4	0.9792 (9)	−0.0350 (9)	0.3519 (17)	0.054 (4)*
C5	0.86265 (7)	−0.04874 (8)	0.32415 (15)	0.0428 (3)
H5	0.8635 (8)	−0.1047 (9)	0.3472 (16)	0.049 (4)*
C6	0.79178 (6)	−0.01189 (6)	0.29224 (13)	0.0337 (2)
C7	0.67194 (6)	−0.03881 (6)	0.34201 (12)	0.0306 (2)
C8	0.67410 (7)	0.03717 (6)	0.41964 (13)	0.0351 (2)
H8A	0.6343 (7)	0.0428 (7)	0.4751 (15)	0.038 (3)*
H8B	0.7249 (8)	0.0453 (8)	0.4914 (15)	0.043 (4)*
C9	0.66091 (6)	0.09902 (6)	0.30295 (13)	0.0349 (2)
C10	0.70987 (8)	0.17555 (6)	0.13187 (14)	0.0371 (3)
H10A	0.7430 (8)	0.1719 (8)	0.0700 (16)	0.042 (4)*
H10B	0.6579 (9)	0.1776 (9)	0.0709 (17)	0.053 (4)*
C11	0.72781 (7)	0.24848 (6)	0.22292 (13)	0.0351 (2)
C12	0.60495 (6)	−0.08991 (6)	0.33007 (13)	0.0336 (2)
C13	0.58545 (8)	−0.14086 (8)	0.21200 (16)	0.0478 (3)
H13	0.6142 (9)	−0.1415 (9)	0.1355 (18)	0.058 (4)*
C14	0.52507 (9)	−0.19148 (9)	0.2063 (2)	0.0638 (4)
H14	0.5166 (13)	−0.2278 (14)	0.127 (3)	0.103 (7)*
C15	0.48451 (9)	−0.19212 (9)	0.3147 (2)	0.0627 (4)
H15	0.4416 (10)	−0.2285 (10)	0.3082 (19)	0.068 (5)*
C16	0.50222 (8)	−0.14105 (8)	0.43070 (19)	0.0527 (4)
H16	0.4772 (11)	−0.1397 (11)	0.505 (2)	0.075 (5)*
C17	0.56172 (7)	−0.08953 (7)	0.43804 (15)	0.0410 (3)
H17	0.5723 (8)	−0.0524 (10)	0.5231 (18)	0.056 (4)*

O4	0.90968 (5)	0.61482 (6)	0.27642 (12)	0.0522 (3)
O5	0.81149 (7)	0.75579 (5)	0.35900 (11)	0.0582 (3)
O6	0.74537 (7)	0.80350 (5)	0.14535 (11)	0.0572 (3)
H6	0.7438	0.8422	0.1948	0.086*
N3	0.78152 (5)	0.60652 (5)	0.25031 (11)	0.0330 (2)
N4	0.76233 (5)	0.43981 (5)	0.28495 (10)	0.0323 (2)
C18	0.71694 (6)	0.57473 (6)	0.29305 (12)	0.0308 (2)
C19	0.65708 (7)	0.62319 (7)	0.30917 (14)	0.0408 (3)
H19	0.6625 (8)	0.6793 (9)	0.3044 (16)	0.050 (4)*
C20	0.59079 (7)	0.59410 (8)	0.33805 (16)	0.0477 (3)
H20	0.5497 (10)	0.6274 (10)	0.3508 (18)	0.064 (5)*
C21	0.58232 (7)	0.51563 (9)	0.35311 (16)	0.0492 (3)
H21	0.5326 (8)	0.4907 (9)	0.3709 (16)	0.047 (4)*
C22	0.64072 (7)	0.46703 (7)	0.33788 (14)	0.0417 (3)
H22	0.6367 (7)	0.4087 (8)	0.3431 (15)	0.044 (4)*
C23	0.70850 (6)	0.49519 (6)	0.30834 (12)	0.0319 (2)
C24	0.83437 (6)	0.45013 (6)	0.33622 (12)	0.0320 (2)
C25	0.86337 (7)	0.52106 (6)	0.42453 (13)	0.0361 (2)
H25A	0.9180 (8)	0.5147 (8)	0.4696 (16)	0.044 (4)*
H25B	0.8342 (8)	0.5321 (8)	0.4972 (16)	0.043 (4)*
C26	0.85536 (6)	0.58557 (6)	0.31257 (14)	0.0352 (2)
C27	0.77099 (8)	0.67215 (6)	0.15010 (14)	0.0374 (3)
H27A	0.8080 (8)	0.6687 (8)	0.0928 (16)	0.045 (4)*
H27B	0.7230 (8)	0.6712 (8)	0.0886 (15)	0.040 (4)*
C28	0.77890 (7)	0.74808 (6)	0.23226 (14)	0.0370 (3)
C29	0.88927 (6)	0.39373 (6)	0.30039 (13)	0.0343 (2)
C30	0.95522 (7)	0.37419 (7)	0.40601 (14)	0.0399 (3)
H30	0.9649 (8)	0.3990 (9)	0.5045 (18)	0.053 (4)*
C31	1.00541 (8)	0.32027 (8)	0.37314 (17)	0.0473 (3)
H31	1.0518 (9)	0.3082 (9)	0.4463 (16)	0.049 (4)*
C32	0.99114 (8)	0.28555 (8)	0.23510 (18)	0.0522 (3)
H32	1.0269 (10)	0.2474 (10)	0.214 (2)	0.070 (5)*
C33	0.92654 (8)	0.30505 (8)	0.12984 (17)	0.0506 (3)
H33	0.9169 (9)	0.2840 (11)	0.035 (2)	0.062 (5)*
C34	0.87589 (7)	0.35883 (7)	0.16167 (14)	0.0422 (3)
H34	0.8301 (9)	0.3742 (9)	0.0780 (17)	0.058 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0432 (5)	0.0407 (5)	0.0819 (7)	0.0120 (4)	0.0244 (5)	0.0092 (5)
O2	0.0514 (5)	0.0230 (4)	0.0563 (5)	0.0006 (3)	0.0041 (4)	−0.0014 (3)
O3	0.0790 (7)	0.0336 (5)	0.0432 (5)	−0.0009 (4)	0.0113 (5)	−0.0035 (4)
N1	0.0369 (5)	0.0214 (4)	0.0453 (5)	0.0001 (3)	0.0157 (4)	0.0017 (4)
N2	0.0356 (5)	0.0217 (4)	0.0458 (5)	−0.0001 (3)	0.0127 (4)	0.0007 (4)
C1	0.0348 (5)	0.0251 (5)	0.0405 (6)	−0.0018 (4)	0.0146 (4)	−0.0031 (4)
C2	0.0420 (7)	0.0348 (6)	0.0577 (8)	−0.0076 (5)	0.0239 (6)	−0.0039 (5)
C3	0.0373 (7)	0.0547 (8)	0.0625 (8)	−0.0112 (6)	0.0218 (6)	−0.0108 (6)

C4	0.0327 (6)	0.0549 (8)	0.0603 (8)	0.0057 (6)	0.0140 (6)	−0.0049 (6)
C5	0.0394 (6)	0.0369 (6)	0.0542 (7)	0.0066 (5)	0.0150 (5)	0.0007 (5)
C6	0.0340 (5)	0.0267 (5)	0.0428 (6)	−0.0004 (4)	0.0138 (5)	−0.0024 (4)
C7	0.0351 (5)	0.0225 (5)	0.0346 (5)	−0.0007 (4)	0.0087 (4)	0.0028 (4)
C8	0.0398 (6)	0.0282 (5)	0.0409 (6)	−0.0046 (4)	0.0167 (5)	−0.0039 (4)
C9	0.0344 (6)	0.0230 (5)	0.0497 (6)	−0.0012 (4)	0.0142 (5)	−0.0036 (4)
C10	0.0488 (7)	0.0226 (5)	0.0414 (6)	−0.0020 (4)	0.0134 (5)	0.0008 (4)
C11	0.0400 (6)	0.0243 (5)	0.0436 (6)	−0.0009 (4)	0.0147 (5)	0.0002 (4)
C12	0.0325 (5)	0.0254 (5)	0.0428 (6)	−0.0014 (4)	0.0078 (4)	0.0045 (4)
C13	0.0465 (7)	0.0399 (7)	0.0572 (8)	−0.0055 (5)	0.0120 (6)	−0.0107 (6)
C14	0.0528 (9)	0.0442 (8)	0.0908 (12)	−0.0123 (6)	0.0087 (8)	−0.0202 (8)
C15	0.0391 (7)	0.0390 (7)	0.1101 (14)	−0.0085 (6)	0.0165 (8)	0.0043 (8)
C16	0.0417 (7)	0.0420 (7)	0.0798 (10)	−0.0007 (5)	0.0249 (7)	0.0154 (7)
C17	0.0395 (6)	0.0357 (6)	0.0499 (7)	−0.0009 (5)	0.0141 (5)	0.0078 (5)
O4	0.0384 (5)	0.0451 (5)	0.0777 (7)	−0.0076 (4)	0.0225 (5)	0.0028 (5)
O5	0.0853 (8)	0.0354 (5)	0.0489 (5)	0.0014 (5)	0.0047 (5)	−0.0069 (4)
O6	0.0824 (7)	0.0286 (4)	0.0572 (6)	0.0155 (5)	0.0082 (5)	−0.0022 (4)
N3	0.0339 (5)	0.0229 (4)	0.0435 (5)	−0.0003 (3)	0.0113 (4)	0.0007 (4)
N4	0.0320 (5)	0.0222 (4)	0.0422 (5)	−0.0006 (3)	0.0073 (4)	−0.0005 (3)
C18	0.0293 (5)	0.0269 (5)	0.0363 (5)	−0.0009 (4)	0.0076 (4)	−0.0039 (4)
C19	0.0375 (6)	0.0332 (6)	0.0516 (7)	0.0044 (5)	0.0098 (5)	−0.0073 (5)
C20	0.0351 (6)	0.0536 (8)	0.0558 (8)	0.0071 (6)	0.0134 (6)	−0.0111 (6)
C21	0.0349 (6)	0.0585 (8)	0.0574 (8)	−0.0066 (6)	0.0175 (6)	−0.0038 (6)
C22	0.0367 (6)	0.0379 (6)	0.0508 (7)	−0.0081 (5)	0.0108 (5)	0.0003 (5)
C23	0.0307 (5)	0.0277 (5)	0.0368 (5)	−0.0021 (4)	0.0066 (4)	−0.0029 (4)
C24	0.0330 (5)	0.0247 (5)	0.0374 (5)	−0.0002 (4)	0.0060 (4)	0.0017 (4)
C25	0.0333 (6)	0.0304 (5)	0.0420 (6)	−0.0012 (4)	0.0028 (5)	−0.0040 (4)
C26	0.0317 (5)	0.0265 (5)	0.0486 (6)	−0.0037 (4)	0.0113 (5)	−0.0060 (4)
C27	0.0477 (7)	0.0244 (5)	0.0407 (6)	0.0005 (4)	0.0115 (5)	0.0005 (4)
C28	0.0442 (6)	0.0246 (5)	0.0453 (6)	0.0000 (4)	0.0167 (5)	−0.0006 (4)
C29	0.0325 (5)	0.0253 (5)	0.0455 (6)	0.0008 (4)	0.0098 (5)	0.0031 (4)
C30	0.0370 (6)	0.0347 (6)	0.0468 (7)	0.0028 (5)	0.0067 (5)	0.0025 (5)
C31	0.0379 (7)	0.0410 (7)	0.0612 (8)	0.0079 (5)	0.0070 (6)	0.0076 (6)
C32	0.0493 (8)	0.0383 (7)	0.0730 (9)	0.0120 (6)	0.0218 (7)	0.0007 (6)
C33	0.0529 (8)	0.0469 (8)	0.0533 (8)	0.0097 (6)	0.0148 (6)	−0.0072 (6)
C34	0.0410 (7)	0.0389 (6)	0.0464 (7)	0.0058 (5)	0.0090 (5)	−0.0013 (5)

Geometric parameters (Å, º)

O1—C9	1.2162 (14)	O4—C26	1.2127 (14)
O2—C11	1.3157 (14)	O5—C28	1.1952 (16)
O2—H2A	0.8200	O6—C28	1.3141 (15)
O3—C11	1.1996 (15)	O6—H6	0.8200
N1—C9	1.3737 (14)	N3—C26	1.3732 (14)
N1—C1	1.4197 (14)	N3—C18	1.4197 (14)
N1—C10	1.4593 (14)	N3—C27	1.4582 (14)
N2—C7	1.2868 (14)	N4—C24	1.2885 (14)
N2—C6	1.4159 (14)	N4—C23	1.4175 (14)

C1—C2	1.3983 (16)	C18—C19	1.4022 (16)
C1—C6	1.4094 (15)	C18—C23	1.4060 (15)
C2—C3	1.382 (2)	C19—C20	1.3750 (19)
C2—H2	0.967 (17)	C19—H19	0.986 (16)
C3—C4	1.382 (2)	C20—C21	1.387 (2)
C3—H3	0.980 (16)	C20—H20	0.967 (17)
C4—C5	1.3808 (19)	C21—C22	1.3807 (19)
C4—H4	0.989 (16)	C21—H21	1.039 (15)
C5—C6	1.3984 (16)	C22—C23	1.3959 (16)
C5—H5	0.999 (15)	C22—H22	1.021 (15)
C7—C12	1.4823 (15)	C24—C29	1.4822 (15)
C7—C8	1.5034 (15)	C24—C25	1.5097 (15)
C8—C9	1.5066 (16)	C25—C26	1.5139 (17)
C8—H8A	0.975 (14)	C25—H25A	0.983 (14)
C8—H8B	1.012 (14)	C25—H25B	0.959 (15)
C10—C11	1.5198 (15)	C27—C28	1.5176 (15)
C10—H10A	0.916 (14)	C27—H27A	0.941 (15)
C10—H10B	0.978 (15)	C27—H27B	0.921 (14)
C12—C13	1.3906 (17)	C29—C34	1.3915 (17)
C12—C17	1.3952 (17)	C29—C30	1.3995 (16)
C13—C14	1.391 (2)	C30—C31	1.3832 (18)
C13—H13	0.964 (16)	C30—H30	0.988 (16)
C14—C15	1.365 (3)	C31—C32	1.384 (2)
C14—H14	0.96 (3)	C31—H31	0.972 (15)
C15—C16	1.376 (2)	C32—C33	1.381 (2)
C15—H15	0.991 (18)	C32—H32	0.974 (19)
C16—C17	1.3870 (18)	C33—C34	1.3838 (18)
C16—H16	0.901 (19)	C33—H33	0.933 (18)
C17—H17	1.004 (17)	C34—H34	1.031 (16)

C11—O2—H2A	109.5	C28—O6—H6	109.5
C9—N1—C1	123.54 (9)	C26—N3—C18	123.59 (9)
C9—N1—C10	114.51 (9)	C26—N3—C27	116.85 (10)
C1—N1—C10	120.19 (9)	C18—N3—C27	119.02 (9)
C7—N2—C6	120.58 (9)	C24—N4—C23	120.87 (9)
C2—C1—C6	118.21 (10)	C19—C18—C23	118.77 (10)
C2—C1—N1	119.56 (10)	C19—C18—N3	119.26 (10)
C6—C1—N1	122.20 (9)	C23—C18—N3	121.78 (9)
C3—C2—C1	121.29 (12)	C20—C19—C18	121.14 (12)
C3—C2—H2	116.4 (9)	C20—C19—H19	118.2 (9)
C1—C2—H2	122.3 (9)	C18—C19—H19	120.6 (9)
C4—C3—C2	120.36 (12)	C19—C20—C21	120.18 (12)
C4—C3—H3	118.0 (10)	C19—C20—H20	121.4 (10)
C2—C3—H3	121.6 (10)	C21—C20—H20	118.4 (10)
C5—C4—C3	119.48 (12)	C22—C21—C20	119.50 (12)
C5—C4—H4	120.6 (9)	C22—C21—H21	117.3 (8)
C3—C4—H4	120.0 (9)	C20—C21—H21	123.1 (8)
C4—C5—C6	121.10 (12)	C21—C22—C23	121.36 (12)

C4—C5—H5	121.2 (8)	C21—C22—H22	122.9 (8)
C6—C5—H5	117.7 (8)	C23—C22—H22	115.7 (8)
C5—C6—C1	119.55 (10)	C22—C23—C18	119.04 (10)
C5—C6—N2	115.48 (10)	C22—C23—N4	116.45 (10)
C1—C6—N2	124.88 (10)	C18—C23—N4	124.34 (9)
N2—C7—C12	119.32 (10)	N4—C24—C29	119.33 (10)
N2—C7—C8	120.71 (10)	N4—C24—C25	120.72 (10)
C12—C7—C8	119.97 (10)	C29—C24—C25	119.86 (10)
C7—C8—C9	107.81 (9)	C24—C25—C26	105.68 (9)
C7—C8—H8A	113.3 (8)	C24—C25—H25A	109.1 (8)
C9—C8—H8A	106.8 (8)	C26—C25—H25A	107.9 (8)
C7—C8—H8B	111.1 (8)	C24—C25—H25B	111.6 (9)
C9—C8—H8B	110.1 (8)	C26—C25—H25B	110.2 (9)
H8A—C8—H8B	107.6 (11)	H25A—C25—H25B	112.1 (12)
O1—C9—N1	121.89 (11)	O4—C26—N3	122.33 (11)
O1—C9—C8	122.57 (11)	O4—C26—C25	122.79 (11)
N1—C9—C8	115.54 (10)	N3—C26—C25	114.83 (10)
N1—C10—C11	111.34 (10)	N3—C27—C28	112.49 (10)
N1—C10—H10A	109.3 (9)	N3—C27—H27A	107.5 (9)
C11—C10—H10A	108.5 (9)	C28—C27—H27A	109.8 (9)
N1—C10—H10B	109.2 (9)	N3—C27—H27B	110.5 (9)
C11—C10—H10B	110.3 (9)	C28—C27—H27B	107.1 (9)
H10A—C10—H10B	108.2 (13)	H27A—C27—H27B	109.5 (12)
O3—C11—O2	124.60 (10)	O5—C28—O6	125.06 (11)
O3—C11—C10	123.72 (10)	O5—C28—C27	124.04 (11)
O2—C11—C10	111.66 (10)	O6—C28—C27	110.90 (10)
C13—C12—C17	118.80 (11)	C34—C29—C30	118.83 (11)
C13—C12—C7	120.07 (11)	C34—C29—C24	120.63 (10)
C17—C12—C7	121.11 (11)	C30—C29—C24	120.54 (10)
C12—C13—C14	119.55 (14)	C31—C30—C29	120.31 (12)
C12—C13—H13	120.0 (10)	C31—C30—H30	120.7 (9)
C14—C13—H13	120.4 (10)	C29—C30—H30	119.0 (9)
C15—C14—C13	121.18 (15)	C30—C31—C32	120.29 (12)
C15—C14—H14	122.6 (14)	C30—C31—H31	119.6 (9)
C13—C14—H14	116.1 (14)	C32—C31—H31	120.1 (9)
C14—C15—C16	119.92 (13)	C33—C32—C31	119.74 (13)
C14—C15—H15	119.8 (10)	C33—C32—H32	121.2 (11)
C16—C15—H15	120.3 (10)	C31—C32—H32	119.1 (11)
C15—C16—C17	119.95 (15)	C32—C33—C34	120.44 (13)
C15—C16—H16	122.8 (12)	C32—C33—H33	121.2 (10)
C17—C16—H16	117.2 (12)	C34—C33—H33	118.4 (10)
C16—C17—C12	120.56 (13)	C33—C34—C29	120.39 (12)
C16—C17—H17	117.7 (9)	C33—C34—H34	118.0 (9)
C12—C17—H17	121.7 (9)	C29—C34—H34	121.5 (9)
C9—N1—C1—C2	−137.08 (12)	C26—N3—C18—C19	137.80 (11)
C10—N1—C1—C2	26.96 (16)	C27—N3—C18—C19	−33.44 (15)
C9—N1—C1—C6	45.09 (16)	C26—N3—C18—C23	−47.32 (16)

C10—N1—C1—C6	−150.87 (11)	C27—N3—C18—C23	141.44 (11)
C6—C1—C2—C3	−1.79 (18)	C23—C18—C19—C20	−0.59 (18)
N1—C1—C2—C3	−179.70 (12)	N3—C18—C19—C20	174.44 (11)
C1—C2—C3—C4	0.9 (2)	C18—C19—C20—C21	0.6 (2)
C2—C3—C4—C5	0.7 (2)	C19—C20—C21—C22	−0.4 (2)
C3—C4—C5—C6	−1.4 (2)	C20—C21—C22—C23	0.3 (2)
C4—C5—C6—C1	0.39 (19)	C21—C22—C23—C18	−0.37 (18)
C4—C5—C6—N2	177.02 (12)	C21—C22—C23—N4	−175.85 (12)
C2—C1—C6—C5	1.16 (17)	C19—C18—C23—C22	0.49 (16)
N1—C1—C6—C5	179.02 (11)	N3—C18—C23—C22	−174.42 (10)
C2—C1—C6—N2	−175.12 (11)	C19—C18—C23—N4	175.58 (10)
N1—C1—C6—N2	2.73 (18)	N3—C18—C23—N4	0.67 (17)
C7—N2—C6—C5	138.67 (11)	C24—N4—C23—C22	−141.09 (11)
C7—N2—C6—C1	−44.91 (17)	C24—N4—C23—C18	43.70 (16)
C6—N2—C7—C12	178.79 (9)	C23—N4—C24—C29	−175.62 (10)
C6—N2—C7—C8	−0.99 (16)	C23—N4—C24—C25	0.86 (16)
N2—C7—C8—C9	73.67 (13)	N4—C24—C25—C26	−74.56 (13)
C12—C7—C8—C9	−106.11 (11)	C29—C24—C25—C26	101.89 (11)
C1—N1—C9—O1	175.41 (11)	C18—N3—C26—O4	−178.32 (11)
C10—N1—C9—O1	10.55 (16)	C27—N3—C26—O4	−6.90 (16)
C1—N1—C9—C8	−5.74 (15)	C18—N3—C26—C25	4.16 (15)
C10—N1—C9—C8	−170.60 (9)	C27—N3—C26—C25	175.58 (9)
C7—C8—C9—O1	111.79 (13)	C24—C25—C26—O4	−108.08 (13)
C7—C8—C9—N1	−67.04 (12)	C24—C25—C26—N3	69.43 (12)
C9—N1—C10—C11	72.77 (13)	C26—N3—C27—C28	−81.33 (13)
C1—N1—C10—C11	−92.65 (13)	C18—N3—C27—C28	90.50 (13)
N1—C10—C11—O3	17.87 (18)	N3—C27—C28—O5	21.02 (18)
N1—C10—C11—O2	−163.44 (10)	N3—C27—C28—O6	−159.08 (11)
N2—C7—C12—C13	−27.62 (16)	N4—C24—C29—C34	36.54 (16)
C8—C7—C12—C13	152.16 (11)	C25—C24—C29—C34	−139.96 (12)
N2—C7—C12—C17	150.59 (11)	N4—C24—C29—C30	−142.93 (11)
C8—C7—C12—C17	−29.63 (15)	C25—C24—C29—C30	40.57 (15)
C17—C12—C13—C14	−1.80 (19)	C34—C29—C30—C31	−0.92 (18)
C7—C12—C13—C14	176.45 (13)	C24—C29—C30—C31	178.56 (11)
C12—C13—C14—C15	0.0 (2)	C29—C30—C31—C32	0.5 (2)
C13—C14—C15—C16	1.3 (3)	C30—C31—C32—C33	0.1 (2)
C14—C15—C16—C17	−0.6 (2)	C31—C32—C33—C34	−0.2 (2)
C15—C16—C17—C12	−1.2 (2)	C32—C33—C34—C29	−0.2 (2)
C13—C12—C17—C16	2.43 (18)	C30—C29—C34—C33	0.81 (19)
C7—C12—C17—C16	−175.80 (11)	C24—C29—C34—C33	−178.67 (12)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C18–C22 and C29–C34 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N4	0.82	1.90	2.7161 (12)	176
O6—H6···N2^{i}	0.82	1.95	2.7585 (13)	171

C4—H4···*Cg*2^{ii}	0.989 (16)	2.97 (2)	3.7650 (16)	139 (1)
C17—H17···*Cg*1^{iii}	1.004 (17)	2.82 (2)	3.6431 (14)	140 (1)

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