IUCrData

ISSN 2414-3146

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

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

[a]Heterocyclic Organic Chemistry Laboratory URAC 21, Faculty of Sciences, Mohammed V University in Rabat, Av Ibn Battouta, BP 1014 Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: sebhaoui.jihad@gmail.com

Received 12 October 2016
Accepted 23 October 2016

Edited by S. Bernès, Benemérita Universidad Autónoma de Puebla, México

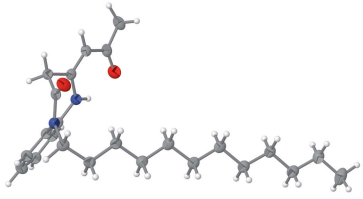
Keywords: crystal structure; benzodiazepine; hydrogen bonding; bilayer.

CCDC reference: 1511185

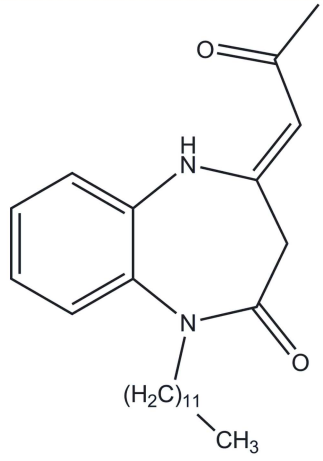
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{24}H_{36}N_2O_2$, the orientation of the 2-oxopropylidene substituent is determined by the formation of an intramolecular N—H···O hydrogen bond. The benzodiazepine seven-membered ring adopts a slightly twisted boat conformation. The molecules pack in a bilayer fashion with the dodecyl chains intercalated to form the inner portion, and the benzodiazepine moieties on the outer surfaces.

3D view



Chemical scheme



Structure description

1,5-Benzodiazepine derivatives are an important class of heterocyclic compounds with versatile biological actions. They are used as anticonvulsivants, myorelaxants (Rudolph *et al.*, 1999), anxiolytics, hypnotics and sedatives (Zellou *et al.*, 1999). The commercial applications of these compounds as dyes for acrylic fibres and in photography have also been reported (Mauget-Fa *et al.*, 2001; Bishop *et al.*, 2007). Two published works indicate that 2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepine derivatives carrying carboxamide substituents are potentially important as therapeutic and prophylactic agents for diabetes, diabetic nephropathy, or glomerulosclerosis (Finch *et al.*, 1996; Ohtake *et al.*, 2007).

In the title molecule, the seven-membered ring adopts a slightly twisted boat conformation. An analysis of the conformation (Cremer & Pople, 1975) yielded the puckering parameters q_2 = 0.893 (2), q_3 = 0.255 (2) Å, φ_2 = 18.2 (1) and φ_3 = 130.0°. The overall puckering amplitude is 0.916 (2) Å. The dodecyl chain is oriented approximately over the bicyclic portion (Fig. 1), while the orientation of the 2-oxopropylidene substituent is determined by the intramolecular hydrogen bond N1—H1···O2 (Table 1 and Fig. 1).

In the crystal, the dodecyl chains intercalate to form the hydrophobic portion of a bilayer, with the more polar bicyclic portions forming the outer faces (Fig. 2). Weak C5—

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.88 (2)	1.94 (2)	2.637 (2)	136 (2)
C5—H5···O1^{i}	0.98 (2)	2.54 (2)	3.500 (2)	165.6 (17)
C2—H2···O2^{ii}	0.97 (2)	2.48 (3)	3.276 (2)	139.4 (19)

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

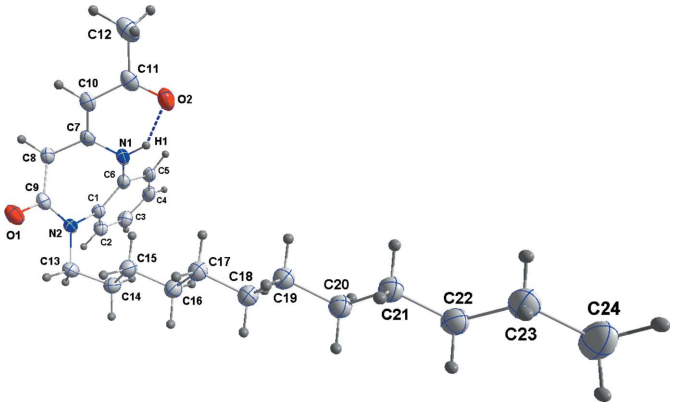


Figure 1
The title molecule with labelling scheme and 50% probability ellipsoids. The intramolecular N—H···O hydrogen bond is shown as a dotted line.

H5···O1^{i} and C2—H2···O2^{ii} [symmetry codes: (i) $x, y-1, z$; (ii) $x-1, y+1, z$] hydrogen bonds help in establishing the surfaces of the bilayer (Table 1 and Fig. 3).

Synthesis and crystallization

A mixture of (4*Z*)-2-oxopropylidene-1,5-benzodiazepin-2-one (0.01 mol), K_2CO_3 (0.02 mol), dodecane bromide (0.02 mol) and tetra-*n*-butylammonium bromide (0.001 mol) in dimethylformamide (60 ml) was stirred at room temperature for 48 h. The solution was filtered by suction filtration. The solvent was removed under reduced pressure. The residue was chromatographed on a silica-gel column using a mixture of hexane and ethyl acetate (80:20 *v*/*v*) as eluent, to afford the title compound as colourless crystals.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{36}N_2O_2$
M_r	384.55
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	8.2589 (9), 8.3715 (9), 18.097 (2)
α, β, γ (°)	87.327 (6), 82.242 (6), 63.248 (7)
V (Å^3)	1106.9 (2)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.57
Crystal size (mm)	0.16 × 0.11 × 0.06
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan *SADABS* (Bruker, 2016)
T_{min}, T_{max}	0.84, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8388, 4075, 3242
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.138, 1.07
No. of reflections	4075
No. of parameters	397
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.27

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

Refinement

Crystal and refinement details are presented in Table 2. All H atoms were found in difference maps and refined with free coordinates and U_{iso} parameters.

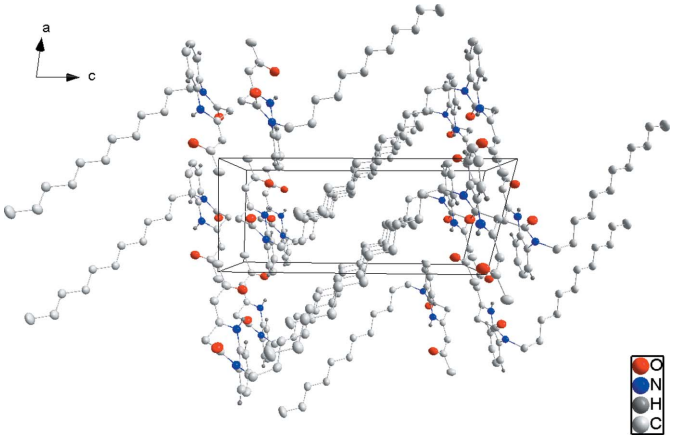


Figure 2
Packing viewed along the *b* axis showing the intercalation of the alkyl chains.

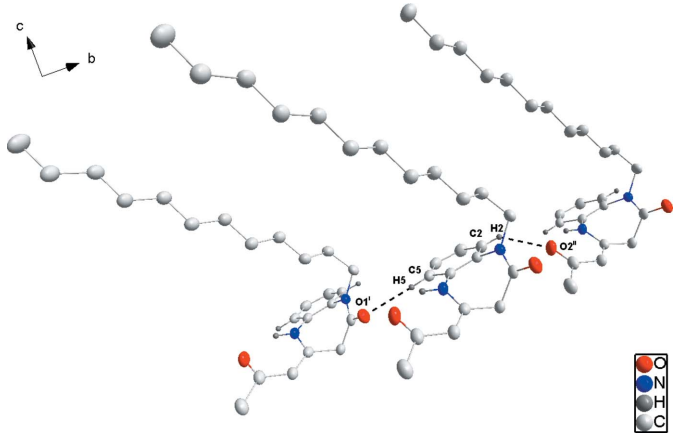


Figure 3
Detail of the intermolecular C—H···O hydrogen bonding [symmetry codes: (i) $x, y-1, z$; (ii) $x-1, y+1, z$].

Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

References

Bishop, S. C., Lerch, M. & McCord, B. R. (2007). *J. Chromatogr. A*, **1154**, 481–484.
Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2016). *APEX3*, *SAINT*, *SADABS* and *SHELXTL*, Bruker AXS, Inc., Madison, WI.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Finch, H., Shah, P. & Carr, R. A. E. (1996). US Patent 5585376 A.
Mauget-Fa, M., Quaranta, M., Francoz, N. & BenEzra, D. (2001). *Graefes Arch. Clin. Exp. Ophthalmol.* **239**, 501–508.
Ohtake, Y., Fukaya, Y., Mihara, Y., Oishi, T., Takashima, Y., Shoji, T. & Morizono, D. (2007). EP Patent 1820799 A1.
Rudolph, U., Crestani, F., Benke, D., Brünig, I., Benson, J. A., Fritschy, J.-M., Martin, J. R., Bluethmann, H. & Möhler, H. (1999). *Nature*, **401**, 796–800.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Zellou, A., Keita, A., Cherrah, Y., Essassi, E. M. & Hassar, M. (1999). *Therapie*, **54**, 645–649.

full crystallographic data

IUCrData (2016). **1**, x161696 [https://doi.org/10.1107/S2414314616016965]

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

Jihad Sebhaoui, Youness El Bakri, El Mokhtar Essassi and Joel T. Mague

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

Crystal data

$C_{24}H_{36}N_2O_2$
M_r = 384.55
Triclinic, $P\overline{1}$
a = 8.2589 (9) Å
b = 8.3715 (9) Å
c = 18.097 (2) Å
α = 87.327 (6)°
β = 82.242 (6)°
γ = 63.248 (7)°
V = 1106.9 (2) Å^3
Z = 2
$F(000)$ = 420
D_x = 1.154 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5933 reflections
θ = 2.5–71.6°
μ = 0.57 mm^{-1}
T = 150 K
Block, colourless
0.16 × 0.11 × 0.06 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} = 0.84, T_{max} = 0.96
8388 measured reflections
4075 independent reflections
3242 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 71.6°, θ_{min} = 4.9°
h = −10→10
k = −10→10
l = −20→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.138
S = 1.07
4075 reflections
397 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
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.49804 (17)	0.95715 (16)	0.11351 (8)	0.0363 (3)

O2	0.85523 (18)	0.18662 (16)	0.11278 (8)	0.0419 (3)
N1	0.5318 (2)	0.46760 (19)	0.12365 (8)	0.0279 (3)
H1	0.600 (3)	0.356 (3)	0.1351 (13)	0.052 (7)*
N2	0.30375 (19)	0.84404 (17)	0.16331 (8)	0.0260 (3)
C1	0.2303 (2)	0.7192 (2)	0.16034 (9)	0.0265 (4)
C2	0.0418 (2)	0.7796 (2)	0.17719 (10)	0.0328 (4)
H2	−0.037 (3)	0.906 (3)	0.1849 (14)	0.055 (7)*
C3	−0.0324 (3)	0.6602 (3)	0.18364 (11)	0.0378 (4)
H3	−0.166 (3)	0.700 (3)	0.1962 (11)	0.032 (5)*
C4	0.0812 (3)	0.4774 (3)	0.17338 (10)	0.0350 (4)
H4	0.025 (3)	0.395 (3)	0.1791 (12)	0.044 (6)*
C5	0.2664 (3)	0.4168 (2)	0.15358 (10)	0.0310 (4)
H5	0.352 (3)	0.291 (3)	0.1420 (12)	0.039 (5)*
C6	0.3426 (2)	0.5368 (2)	0.14575 (9)	0.0261 (3)
C7	0.6123 (2)	0.5373 (2)	0.06987 (9)	0.0255 (3)
H7	0.844 (3)	0.493 (3)	0.0001 (11)	0.032 (5)*
C8	0.4925 (2)	0.7223 (2)	0.04494 (9)	0.0260 (4)
H8A	0.380 (3)	0.723 (3)	0.0262 (11)	0.037 (5)*
H8B	0.556 (3)	0.759 (3)	0.0042 (11)	0.033 (5)*
C9	0.4323 (2)	0.8528 (2)	0.10986 (9)	0.0259 (4)
C10	0.7909 (2)	0.4437 (2)	0.04016 (10)	0.0277 (4)
C11	0.9060 (2)	0.2644 (2)	0.06113 (10)	0.0315 (4)
C12	1.0942 (3)	0.1678 (3)	0.01860 (13)	0.0393 (5)
H12A	1.181 (4)	0.172 (3)	0.0470 (14)	0.059 (7)*
H12B	1.116 (3)	0.228 (3)	−0.0302 (15)	0.058 (7)*
H12C	1.119 (4)	0.050 (4)	0.0081 (18)	0.087 (10)*
C13	0.2425 (3)	0.9605 (2)	0.23012 (10)	0.0315 (4)
H13A	0.103 (3)	1.037 (3)	0.2358 (12)	0.046 (6)*
H13B	0.305 (3)	1.039 (3)	0.2213 (11)	0.038 (5)*
C14	0.2929 (2)	0.8527 (2)	0.30084 (10)	0.0319 (4)
H14A	0.228 (3)	0.780 (3)	0.3095 (12)	0.041 (6)*
H14B	0.251 (3)	0.935 (3)	0.3452 (13)	0.051 (6)*
C15	0.4971 (2)	0.7328 (2)	0.30015 (10)	0.0317 (4)
H15A	0.544 (3)	0.640 (3)	0.2587 (12)	0.037 (5)*
H15B	0.563 (3)	0.812 (3)	0.2879 (12)	0.040 (5)*
C16	0.5431 (3)	0.6367 (3)	0.37373 (10)	0.0336 (4)
H16A	0.472 (3)	0.571 (3)	0.3864 (12)	0.043 (6)*
H16B	0.498 (3)	0.731 (3)	0.4173 (14)	0.052 (6)*
C17	0.7447 (3)	0.5139 (2)	0.37574 (10)	0.0340 (4)
H17A	0.791 (3)	0.417 (3)	0.3350 (14)	0.052 (6)*
H17B	0.816 (3)	0.585 (3)	0.3631 (12)	0.042 (6)*
C18	0.7845 (3)	0.4253 (3)	0.45129 (10)	0.0346 (4)
H18A	0.707 (3)	0.359 (3)	0.4649 (13)	0.051 (6)*
H18B	0.737 (3)	0.520 (3)	0.4902 (13)	0.047 (6)*
C19	0.9837 (3)	0.3002 (2)	0.45673 (10)	0.0343 (4)
H19A	1.029 (3)	0.202 (3)	0.4173 (13)	0.044 (6)*
H19B	1.058 (3)	0.369 (3)	0.4447 (13)	0.048 (6)*
C20	1.0145 (3)	0.2144 (2)	0.53318 (10)	0.0343 (4)

H20A	0.939 (3)	0.147 (3)	0.5437 (13)	0.049 (6)*
H20B	0.967 (3)	0.312 (3)	0.5731 (13)	0.043 (6)*
C21	1.2120 (3)	0.0869 (2)	0.54133 (10)	0.0338 (4)
H21A	1.259 (3)	−0.011 (3)	0.5017 (13)	0.046 (6)*
H21B	1.287 (3)	0.154 (3)	0.5297 (12)	0.042 (6)*
C22	1.2391 (3)	0.0029 (3)	0.61809 (11)	0.0357 (4)
H22A	1.159 (3)	−0.059 (3)	0.6320 (13)	0.052 (6)*
H22B	1.193 (3)	0.099 (3)	0.6567 (13)	0.048 (6)*
C23	1.4358 (3)	−0.1263 (3)	0.62625 (12)	0.0401 (4)
H23A	1.516 (4)	−0.063 (3)	0.6143 (14)	0.058 (7)*
H23B	1.479 (3)	−0.219 (3)	0.5888 (14)	0.050 (6)*
C24	1.4624 (4)	−0.2082 (3)	0.70285 (14)	0.0550 (6)
H24A	1.600 (4)	−0.300 (4)	0.7042 (16)	0.077 (9)*
H24B	1.426 (4)	−0.117 (4)	0.7445 (16)	0.066 (8)*
H24C	1.388 (4)	−0.273 (4)	0.7164 (17)	0.082 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0366 (7)	0.0284 (6)	0.0495 (8)	−0.0200 (6)	−0.0030 (6)	−0.0033 (5)
O2	0.0400 (8)	0.0286 (7)	0.0529 (9)	−0.0124 (6)	−0.0063 (6)	0.0098 (6)
N1	0.0302 (7)	0.0216 (7)	0.0320 (8)	−0.0118 (6)	−0.0044 (6)	0.0038 (6)
N2	0.0288 (7)	0.0220 (7)	0.0276 (7)	−0.0117 (6)	−0.0037 (5)	−0.0004 (5)
C1	0.0315 (9)	0.0260 (8)	0.0250 (8)	−0.0153 (7)	−0.0049 (6)	0.0019 (6)
C2	0.0326 (9)	0.0310 (9)	0.0352 (10)	−0.0151 (8)	−0.0032 (7)	0.0033 (7)
C3	0.0322 (10)	0.0435 (11)	0.0416 (10)	−0.0213 (9)	−0.0029 (8)	0.0045 (8)
C4	0.0432 (10)	0.0398 (10)	0.0326 (9)	−0.0280 (9)	−0.0059 (7)	0.0045 (8)
C5	0.0424 (10)	0.0290 (9)	0.0271 (9)	−0.0206 (8)	−0.0062 (7)	0.0028 (7)
C6	0.0309 (8)	0.0257 (8)	0.0246 (8)	−0.0147 (7)	−0.0067 (6)	0.0040 (6)
C7	0.0310 (8)	0.0235 (8)	0.0260 (8)	−0.0148 (7)	−0.0068 (6)	0.0000 (6)
C8	0.0271 (8)	0.0230 (8)	0.0276 (8)	−0.0110 (7)	−0.0048 (6)	0.0044 (6)
C9	0.0254 (8)	0.0198 (8)	0.0317 (9)	−0.0087 (7)	−0.0070 (6)	0.0036 (6)
C10	0.0281 (8)	0.0248 (8)	0.0318 (9)	−0.0128 (7)	−0.0064 (7)	0.0021 (7)
C11	0.0310 (9)	0.0257 (8)	0.0397 (10)	−0.0128 (7)	−0.0110 (7)	−0.0001 (7)
C12	0.0296 (10)	0.0303 (10)	0.0570 (13)	−0.0116 (8)	−0.0071 (9)	−0.0034 (9)
C13	0.0363 (10)	0.0244 (8)	0.0317 (9)	−0.0118 (8)	−0.0023 (7)	−0.0039 (7)
C14	0.0331 (9)	0.0338 (9)	0.0296 (9)	−0.0158 (8)	−0.0014 (7)	−0.0037 (7)
C15	0.0336 (9)	0.0332 (9)	0.0285 (9)	−0.0155 (8)	−0.0025 (7)	−0.0013 (7)
C16	0.0355 (10)	0.0354 (9)	0.0305 (9)	−0.0163 (8)	−0.0045 (7)	0.0012 (8)
C17	0.0378 (10)	0.0344 (9)	0.0305 (9)	−0.0166 (9)	−0.0059 (7)	0.0017 (8)
C18	0.0379 (10)	0.0340 (10)	0.0315 (10)	−0.0156 (8)	−0.0056 (7)	0.0013 (8)
C19	0.0396 (10)	0.0347 (10)	0.0303 (9)	−0.0181 (9)	−0.0053 (7)	0.0016 (8)
C20	0.0390 (10)	0.0340 (10)	0.0320 (10)	−0.0179 (9)	−0.0057 (7)	0.0022 (8)
C21	0.0381 (10)	0.0330 (9)	0.0314 (9)	−0.0166 (8)	−0.0067 (7)	0.0037 (8)
C22	0.0431 (11)	0.0350 (10)	0.0327 (10)	−0.0204 (9)	−0.0062 (8)	0.0025 (8)
C23	0.0464 (11)	0.0379 (10)	0.0394 (11)	−0.0200 (9)	−0.0139 (9)	0.0040 (9)
C24	0.0745 (17)	0.0507 (13)	0.0491 (14)	−0.0320 (13)	−0.0288 (12)	0.0158 (11)

Geometric parameters (Å, º)

O1—C9	1.2263 (19)	C14—H14A	0.97 (2)
O2—C11	1.247 (2)	C14—H14B	1.00 (2)
N1—C7	1.360 (2)	C15—C16	1.525 (3)
N1—C6	1.405 (2)	C15—H15A	1.01 (2)
N1—H1	0.88 (2)	C15—H15B	1.03 (2)
N2—C9	1.362 (2)	C16—C17	1.517 (3)
N2—C1	1.4309 (19)	C16—H16A	0.97 (2)
N2—C13	1.473 (2)	C16—H16B	1.05 (2)
C1—C2	1.396 (2)	C17—C18	1.528 (3)
C1—C6	1.400 (2)	C17—H17A	1.02 (2)
C2—C3	1.382 (2)	C17—H17B	1.01 (2)
C2—H2	0.97 (2)	C18—C19	1.515 (3)
C3—C4	1.394 (3)	C18—H18A	1.03 (3)
C3—H3	1.00 (2)	C18—H18B	0.99 (2)
C4—C5	1.376 (3)	C19—C20	1.527 (3)
C4—H4	0.98 (2)	C19—H19A	1.01 (2)
C5—C6	1.399 (2)	C19—H19B	1.01 (2)
C5—H5	0.98 (2)	C20—C21	1.518 (3)
C7—C10	1.365 (2)	C20—H20A	1.01 (2)
C7—C8	1.502 (2)	C20—H20B	1.02 (2)
C8—C9	1.515 (2)	C21—C22	1.525 (3)
C8—H8A	1.03 (2)	C21—H21A	1.01 (2)
C8—H8B	0.96 (2)	C21—H21B	1.01 (2)
C10—C11	1.436 (2)	C22—C23	1.516 (3)
C10—H7	0.97 (2)	C22—H22A	1.01 (2)
C11—C12	1.506 (3)	C22—H22B	0.99 (2)
C12—H12A	0.95 (3)	C23—C24	1.516 (3)
C12—H12B	1.03 (3)	C23—H23A	1.02 (3)
C12—H12C	0.94 (3)	C23—H23B	0.96 (2)
C13—C14	1.525 (3)	C24—H24A	1.05 (3)
C13—H13A	1.03 (2)	C24—H24B	1.02 (3)
C13—H13B	1.00 (2)	C24—H24C	0.99 (3)
C14—C15	1.524 (3)		

C7—N1—C6	125.16 (15)	C14—C15—C16	112.49 (15)
C7—N1—H1	115.3 (16)	C14—C15—H15A	110.6 (12)
C6—N1—H1	117.0 (16)	C16—C15—H15A	108.4 (12)
C9—N2—C1	123.31 (14)	C14—C15—H15B	107.5 (12)
C9—N2—C13	119.43 (13)	C16—C15—H15B	111.2 (12)
C1—N2—C13	117.17 (13)	H15A—C15—H15B	106.5 (16)
C2—C1—C6	119.07 (15)	C17—C16—C15	114.65 (15)
C2—C1—N2	118.86 (15)	C17—C16—H16A	110.3 (13)
C6—C1—N2	121.94 (15)	C15—C16—H16A	110.0 (14)
C3—C2—C1	120.67 (17)	C17—C16—H16B	108.4 (13)
C3—C2—H2	119.9 (15)	C15—C16—H16B	109.6 (13)
C1—C2—H2	119.4 (15)	H16A—C16—H16B	103.3 (18)

C2—C3—C4	120.01 (17)	C16—C17—C18	112.69 (15)
C2—C3—H3	122.0 (11)	C16—C17—H17A	109.8 (14)
C4—C3—H3	117.9 (11)	C18—C17—H17A	109.3 (14)
C5—C4—C3	119.89 (16)	C16—C17—H17B	108.9 (13)
C5—C4—H4	121.9 (13)	C18—C17—H17B	110.0 (13)
C3—C4—H4	118.2 (13)	H17A—C17—H17B	105.9 (18)
C4—C5—C6	120.54 (17)	C19—C18—C17	115.22 (15)
C4—C5—H5	123.4 (12)	C19—C18—H18A	109.9 (13)
C6—C5—H5	116.0 (12)	C17—C18—H18A	109.4 (13)
C5—C6—C1	119.64 (16)	C19—C18—H18B	110.1 (14)
C5—C6—N1	117.97 (15)	C17—C18—H18B	108.6 (14)
C1—C6—N1	122.39 (14)	H18A—C18—H18B	103.0 (18)
N1—C7—C10	121.79 (15)	C18—C19—C20	112.74 (15)
N1—C7—C8	116.00 (14)	C18—C19—H19A	108.9 (13)
C10—C7—C8	122.22 (15)	C20—C19—H19A	109.0 (13)
C7—C8—C9	109.41 (14)	C18—C19—H19B	108.8 (13)
C7—C8—H8A	109.1 (11)	C20—C19—H19B	110.5 (13)
C9—C8—H8A	109.9 (11)	H19A—C19—H19B	106.7 (18)
C7—C8—H8B	111.1 (12)	C21—C20—C19	114.78 (16)
C9—C8—H8B	109.7 (12)	C21—C20—H20A	108.5 (13)
H8A—C8—H8B	107.6 (17)	C19—C20—H20A	108.7 (13)
O1—C9—N2	123.08 (15)	C21—C20—H20B	108.9 (13)
O1—C9—C8	121.36 (15)	C19—C20—H20B	109.2 (12)
N2—C9—C8	115.55 (13)	H20A—C20—H20B	106.5 (17)
C7—C10—C11	123.18 (16)	C20—C21—C22	113.78 (16)
C7—C10—H7	120.6 (12)	C20—C21—H21A	108.8 (13)
C11—C10—H7	116.0 (12)	C22—C21—H21A	109.5 (13)
O2—C11—C10	122.41 (16)	C20—C21—H21B	108.3 (12)
O2—C11—C12	119.32 (17)	C22—C21—H21B	110.3 (12)
C10—C11—C12	118.27 (17)	H21A—C21—H21B	105.8 (17)
C11—C12—H12A	109.1 (15)	C23—C22—C21	113.99 (16)
C11—C12—H12B	114.8 (14)	C23—C22—H22A	109.3 (14)
H12A—C12—H12B	102 (2)	C21—C22—H22A	111.5 (14)
C11—C12—H12C	109.9 (19)	C23—C22—H22B	109.1 (14)
H12A—C12—H12C	112 (2)	C21—C22—H22B	109.3 (13)
H12B—C12—H12C	109 (2)	H22A—C22—H22B	103.0 (18)
N2—C13—C14	111.77 (14)	C24—C23—C22	113.8 (2)
N2—C13—H13A	110.2 (12)	C24—C23—H23A	109.4 (15)
C14—C13—H13A	108.4 (13)	C22—C23—H23A	109.5 (14)
N2—C13—H13B	105.3 (12)	C24—C23—H23B	109.9 (14)
C14—C13—H13B	110.9 (12)	C22—C23—H23B	108.4 (15)
H13A—C13—H13B	110.3 (17)	H23A—C23—H23B	105.5 (19)
C15—C14—C13	114.11 (15)	C23—C24—H24A	110.0 (16)
C15—C14—H14A	108.9 (13)	C23—C24—H24B	113.5 (15)
C13—C14—H14A	110.0 (13)	H24A—C24—H24B	108 (2)
C15—C14—H14B	107.8 (14)	C23—C24—H24C	112.0 (18)
C13—C14—H14B	110.0 (14)	H24A—C24—H24C	108 (2)
H14A—C14—H14B	105.6 (18)	H24B—C24—H24C	106 (2)

C9—N2—C1—C2	−135.43 (17)	C13—N2—C9—O1	−1.8 (3)
C13—N2—C1—C2	48.1 (2)	C1—N2—C9—C8	1.0 (2)
C9—N2—C1—C6	48.7 (2)	C13—N2—C9—C8	177.42 (14)
C13—N2—C1—C6	−127.77 (17)	C7—C8—C9—O1	105.68 (18)
C6—C1—C2—C3	3.7 (3)	C7—C8—C9—N2	−73.53 (18)
N2—C1—C2—C3	−172.29 (16)	N1—C7—C10—C11	1.9 (3)
C1—C2—C3—C4	0.1 (3)	C8—C7—C10—C11	−177.63 (15)
C2—C3—C4—C5	−2.9 (3)	C7—C10—C11—O2	−6.3 (3)
C3—C4—C5—C6	1.9 (3)	C7—C10—C11—C12	173.45 (16)
C4—C5—C6—C1	1.9 (3)	C9—N2—C13—C14	−115.69 (17)
C4—C5—C6—N1	−178.46 (16)	C1—N2—C13—C14	61.0 (2)
C2—C1—C6—C5	−4.6 (2)	N2—C13—C14—C15	58.3 (2)
N2—C1—C6—C5	171.20 (15)	C13—C14—C15—C16	176.23 (14)
C2—C1—C6—N1	175.74 (16)	C14—C15—C16—C17	179.39 (15)
N2—C1—C6—N1	−8.4 (2)	C15—C16—C17—C18	178.22 (15)
C7—N1—C6—C5	133.69 (17)	C16—C17—C18—C19	179.72 (16)
C7—N1—C6—C1	−46.7 (2)	C17—C18—C19—C20	−179.42 (15)
C6—N1—C7—C10	−165.76 (15)	C18—C19—C20—C21	179.65 (15)
C6—N1—C7—C8	13.8 (2)	C19—C20—C21—C22	−179.93 (15)
N1—C7—C8—C9	63.24 (18)	C20—C21—C22—C23	179.33 (16)
C10—C7—C8—C9	−117.19 (17)	C21—C22—C23—C24	179.49 (17)
C1—N2—C9—O1	−178.22 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.88 (2)	1.94 (2)	2.637 (2)	136 (2)
C5—H5···O1^{i}	0.98 (2)	2.54 (2)	3.500 (2)	165.6 (17)
C2—H2···O2^{ii}	0.97 (2)	2.48 (3)	3.276 (2)	139.4 (19)

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