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(*E*)-2-Phenyl-4-styryl-2,3-dihydro-1*H*-1,5-benzodiazepine hemihydrate

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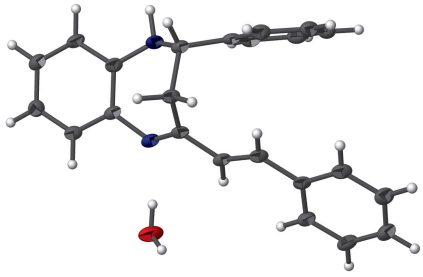
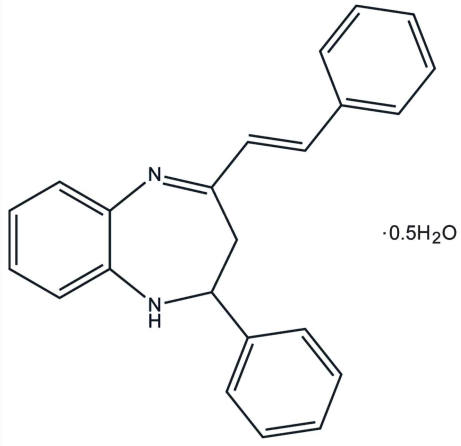
Structural data: full structural data are available from iucrdata.iucr.org

The unit cell contains eight molecules of the title compound, with half a water molecule per main molecule, $C_{23}H_{20}N_2\cdot0.5H_2O$. The seven-membered diazepine ring adopts a twist-boat conformation and makes dihedral angles of 85.08 (7) and 32.79 (7)° with the phenyl and styryl substituents, respectively. In the crystal, the organic molecules are linked by C—H···N hydrogen bonds into chains running along the *b*-axis direction. The water molecule, located on a twofold rotation axis, forms hydrogen bridges, connecting two adjacent chains.

3D view



Chemical scheme



Structure description

Benzodiazepines are heterocyclic compounds that are considered to be 'privileged structures' since they possess a wide range of biological activities. 1,5-Benzodiazepines have found applications in medicine, being one of the most important classes of the therapeutic agents with widespread biological activities. They are commonly used as anti-inflammatory (Bhat & Kumar, 2016), antioxidant (Patil *et al.*, 2015), anticancer (Chen *et al.*, 2014), antimicrobial (El-Gaml *et al.*, 2014) and antiviral (Nyanguile *et al.*, 2008) substances and constitute the backbones of several marketed drugs. 1,5-Benzodiazepines are generally synthesized by the condensation of *o*-phenylenediamine with α,β-unsaturated carbonyl compounds (Claramunt *et al.*, 2006), β-haloketones (Ilango *et al.*, 2013), or with ketones using acidic catalysts (Jeganathan & Pitchumani, 2014) or the microwave irradiation technique, which is critical to enhance the condensation process (Chikhale & Khedekar, 2013). We report here the synthesis and characterization of a new 1,5-benzodiazepine derivative.

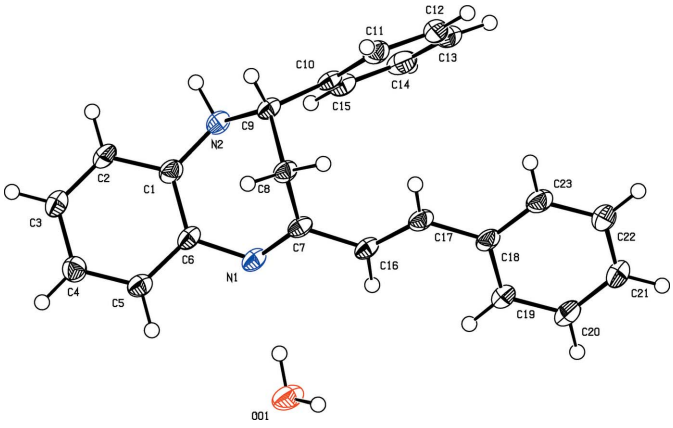


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

The molecular structure of the title compound is illustrated in Fig. 1. In the molecule, which adopts an approximate U shape, the seven-membered diazepine ring displays a twist-boat conformation as indicated by the total puckering amplitude Q_T = 0.9442 (19)°, and spherical polar angle $\theta 2$ = 79.23 (11)°; $\varphi 2$= 118.15 (13)° and $\varphi 3$ = −104.5 (7)°. The benzodiazepine ring system makes dihedral angles of 85.08 (7) and 32.79 (7)° with the phenyl and styryl substituents, respectively.

In the crystal, the organic molecules are linked by C11—H11···N1 hydrogen bonds (Table 1, Fig. 2) into chains running along the *b*-axis direction. The water molecule, located on a twofold rotation axis, forms hydrogen bridges [N1···O01···N1($\frac{1}{2}-x, y, -z$); Fig. 3], connecting two adjacent chains.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O01—H01···N1	0.92 (3)	2.01 (3)	2.9300 (19)	174 (3)
C11—H11···N1^{i}	0.95	2.51	3.426 (3)	161

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{20}N_2{\cdot}0.5H_2O$
M_r	333.42
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	100
a, *b*, *c* (Å)	22.1001 (8), 6.7624 (2), 24.5714 (7)
β (°)	104.835 (3)
V (Å^3)	3549.8 (2)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.58
Crystal size (mm)	0.21 × 0.16 × 0.04
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31210, 3649, 2831
R_{int}	0.148
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.058, 0.172, 1.07
No. of reflections	3649
No. of parameters	240
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.30

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014*(Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

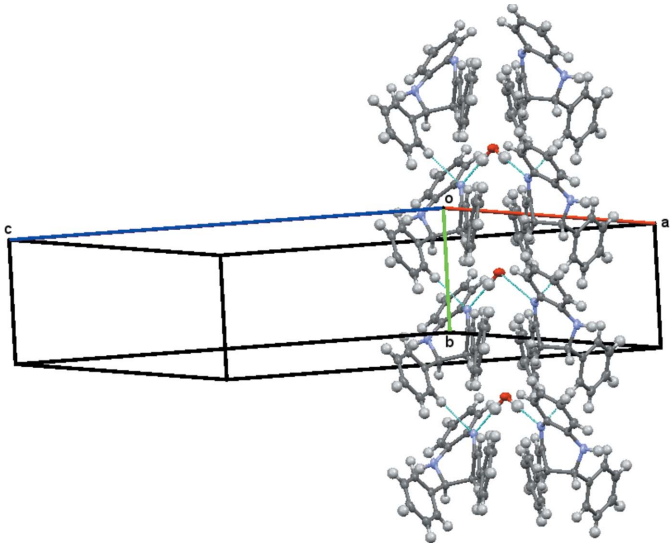


Figure 2
A view of the chains formed by C11—H11···N1 hydrogen bonds (Table 1) and linked by N1···O01···N1 bridges.

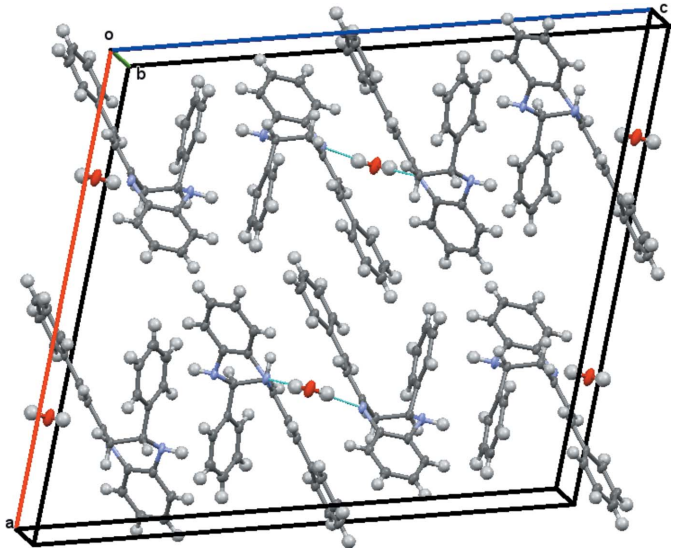


Figure 3
Overall packing of the title compound, showing the N1—O01—N1 bridges.

Synthesis and crystallization

To a solution of 1,7-diphenylhepta-1,6-diene-3,5-dione (0.1 mol) in ethanol (30 ml) a few drops of triethylamine and 1,2-diaminobenzene (0.1 mol) were added. The mixture was heated under reflux for 12 h. The solvent was evaporated. The title compound was isolated by column chromatography on silica gel using hexane/ethyl acetate as eluent. The solid product was recrystallized in ethyl acetate to give crystals of the title compound.

Refinement

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

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full crystallographic data

IUCrData (2017). **2**, x171070 [https://doi.org/10.1107/S2414314617010707]

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(*E*)-2-Phenyl-4-styryl-2,3-dihydro-1*H*-1,5-benzodiazepine hemihydrate

Crystal data

$C_{23}H_{20}N_2 \cdot 0.5H_2O$
$M_r = 333.42$
Monoclinic, *I*2/*a*
$a = 22.1001\ (8)$ Å
$b = 6.7624\ (2)$ Å
$c = 24.5714\ (7)$ Å
$\beta = 104.835\ (3)°$
$V = 3549.8\ (2)$ Å^3
$Z = 8$
$F(000) = 1416$
$D_x = 1.248$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3649 reflections
$\theta = 3.7–74.5°$
$\mu = 0.58$ mm^{-1}
$T = 100$ K
Plate, colourless
0.21 × 0.16 × 0.04 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)

31210 measured reflections

3649 independent reflections
2831 reflections with $I > 2\sigma(I)$
$R_{int} = 0.148$
$\theta_{max} = 74.5°$, $\theta_{min} = 3.7°$
$h = -27\rightarrow27$
$k = -8\rightarrow8$
$l = -30\rightarrow30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.172$
$S = 1.07$
3649 reflections
240 parameters
0 restraints
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0998P)^2 + 0.8126P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00032 (11)

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. All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.99 Å (methylene), 0.98 Å (methyl), 1.0Å (methine) with $U_{iso}(H) = 1.2U_{eq}$(CH and CH_2). The coordinates of H atoms attached to N atoms were freely refined with $U_{iso}(H) = 1.2U_{eq}(N)$ and the H attached to hydroxyl O atoms were fixed geometrically and treated as riding with O—H = 0.84Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O01	0.250000	0.4867 (3)	0.000000	0.0395 (5)
H	0.3281 (13)	0.996 (4)	0.2434 (12)	0.037 (7)*
H01	0.2594 (15)	0.572 (5)	0.0303 (12)	0.051 (8)*
C16	0.20259 (9)	0.9411 (3)	0.04598 (7)	0.0244 (4)
H16	0.184023	0.819984	0.030619	0.029*
N2	0.31472 (8)	0.9651 (2)	0.20337 (6)	0.0282 (4)
N1	0.28863 (8)	0.7563 (2)	0.09599 (6)	0.0249 (4)
C18	0.10669 (9)	1.1185 (3)	−0.00793 (6)	0.0240 (4)
C17	0.16963 (9)	1.1077 (3)	0.03003 (7)	0.0249 (4)
H17	0.188813	1.228920	0.044640	0.030*
C7	0.26467 (9)	0.9315 (3)	0.08503 (6)	0.0227 (4)
C6	0.34457 (9)	0.7303 (3)	0.13831 (7)	0.0231 (4)
C5	0.38511 (10)	0.5796 (3)	0.13048 (7)	0.0249 (4)
H5	0.375753	0.509354	0.095850	0.030*
C10	0.23119 (9)	1.2139 (3)	0.17243 (6)	0.0234 (4)
C21	−0.01405 (11)	1.1536 (3)	−0.07978 (8)	0.0341 (5)
H21	−0.054992	1.165117	−0.103952	0.041*
C1	0.35798 (9)	0.8318 (3)	0.19049 (7)	0.0252 (4)
C9	0.29638 (9)	1.1477 (3)	0.17111 (7)	0.0236 (4)
H9	0.326737	1.254179	0.188115	0.028*
C8	0.29998 (9)	1.1139 (3)	0.11003 (7)	0.0235 (4)
H8A	0.282325	1.230251	0.086914	0.028*
H8B	0.344362	1.100613	0.109275	0.028*
C2	0.41197 (10)	0.7797 (3)	0.23157 (7)	0.0288 (4)
H2	0.421618	0.847780	0.266563	0.035*
C11	0.21281 (10)	1.4043 (3)	0.15406 (7)	0.0283 (4)
H11	0.242354	1.491690	0.144851	0.034*
C4	0.43838 (10)	0.5304 (3)	0.17179 (8)	0.0280 (4)
H4	0.465374	0.428633	0.165373	0.034*
C23	0.07520 (10)	1.2995 (3)	−0.01512 (7)	0.0287 (4)
H23	0.094978	1.412296	0.004770	0.034*
C12	0.15186 (11)	1.4688 (3)	0.14894 (8)	0.0324 (4)
H12	0.139662	1.598047	0.135302	0.039*
C15	0.18828 (10)	1.0901 (3)	0.18831 (7)	0.0271 (4)
H15	0.200434	0.960834	0.201990	0.033*
C22	0.01555 (11)	1.3179 (3)	−0.05075 (8)	0.0321 (4)
H22	−0.004983	1.442502	−0.055280	0.039*
C3	0.45205 (10)	0.6315 (3)	0.22288 (8)	0.0293 (4)
H3	0.488507	0.599417	0.251574	0.035*
C19	0.07606 (10)	0.9550 (3)	−0.03779 (7)	0.0294 (4)

H19	0.096421	0.830136	−0.033653	0.035*
C14	0.12768 (10)	1.1559 (3)	0.18410 (8)	0.0329 (5)
H14	0.098747	1.071108	0.195262	0.039*
C13	0.10878 (10)	1.3435 (3)	0.16385 (8)	0.0344 (5)
H13	0.066951	1.385892	0.160215	0.041*
C20	0.01653 (11)	0.9730 (3)	−0.07325 (8)	0.0357 (5)
H20	−0.003486	0.860635	−0.093225	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O01	0.0638 (16)	0.0241 (9)	0.0237 (9)	0.000	−0.0016 (9)	0.000
C16	0.0288 (10)	0.0296 (9)	0.0128 (6)	−0.0014 (7)	0.0019 (6)	−0.0005 (6)
N2	0.0342 (9)	0.0366 (8)	0.0120 (6)	0.0074 (7)	0.0028 (6)	0.0019 (6)
N1	0.0291 (9)	0.0289 (8)	0.0146 (6)	−0.0020 (6)	0.0014 (6)	0.0009 (5)
C18	0.0281 (10)	0.0324 (9)	0.0113 (7)	−0.0001 (7)	0.0045 (6)	0.0003 (6)
C17	0.0315 (10)	0.0289 (8)	0.0136 (7)	−0.0005 (7)	0.0044 (7)	−0.0003 (6)
C7	0.0284 (10)	0.0280 (8)	0.0116 (7)	−0.0021 (7)	0.0047 (6)	0.0002 (6)
C6	0.0255 (9)	0.0258 (8)	0.0156 (7)	−0.0029 (7)	0.0005 (6)	0.0016 (6)
C5	0.0298 (10)	0.0247 (8)	0.0189 (7)	−0.0023 (7)	0.0041 (7)	−0.0003 (6)
C10	0.0283 (9)	0.0289 (8)	0.0113 (6)	−0.0018 (7)	0.0019 (6)	−0.0037 (6)
C21	0.0316 (11)	0.0427 (11)	0.0227 (8)	0.0045 (9)	−0.0026 (7)	−0.0001 (7)
C1	0.0294 (10)	0.0285 (8)	0.0166 (7)	0.0008 (7)	0.0040 (7)	0.0022 (6)
C9	0.0267 (10)	0.0278 (9)	0.0144 (7)	−0.0011 (7)	0.0019 (6)	−0.0009 (6)
C8	0.0286 (10)	0.0269 (8)	0.0144 (7)	−0.0002 (7)	0.0043 (6)	0.0014 (6)
C2	0.0314 (10)	0.0343 (9)	0.0162 (7)	0.0021 (8)	−0.0021 (7)	−0.0005 (7)
C11	0.0354 (11)	0.0304 (9)	0.0183 (7)	0.0005 (8)	0.0056 (7)	−0.0001 (6)
C4	0.0286 (10)	0.0288 (9)	0.0261 (8)	0.0027 (7)	0.0062 (7)	0.0022 (7)
C23	0.0365 (11)	0.0309 (9)	0.0171 (7)	−0.0006 (8)	0.0042 (7)	−0.0004 (6)
C12	0.0372 (11)	0.0368 (10)	0.0202 (8)	0.0106 (9)	0.0022 (7)	−0.0026 (7)
C15	0.0322 (10)	0.0307 (9)	0.0174 (7)	−0.0034 (8)	0.0047 (7)	−0.0022 (6)
C22	0.0362 (11)	0.0355 (10)	0.0214 (8)	0.0064 (8)	0.0015 (8)	0.0026 (7)
C3	0.0276 (10)	0.0344 (10)	0.0222 (8)	0.0013 (8)	−0.0007 (7)	0.0021 (7)
C19	0.0307 (10)	0.0329 (9)	0.0205 (8)	0.0023 (8)	−0.0007 (7)	−0.0036 (7)
C14	0.0313 (11)	0.0430 (11)	0.0248 (8)	−0.0082 (8)	0.0078 (8)	−0.0098 (8)
C13	0.0287 (11)	0.0474 (12)	0.0247 (8)	0.0044 (9)	0.0023 (7)	−0.0137 (8)
C20	0.0356 (12)	0.0396 (11)	0.0252 (8)	−0.0009 (9)	−0.0044 (8)	−0.0064 (8)

Geometric parameters (Å, º)

O01—H01	0.92 (3)	C9—C8	1.540 (2)
C16—C17	1.344 (3)	C9—H9	1.0000
C16—C7	1.460 (3)	C8—H8A	0.9900
C16—H16	0.9500	C8—H8B	0.9900
N2—C1	1.407 (2)	C2—C3	1.390 (3)
N2—C9	1.467 (2)	C2—H2	0.9500
N2—H	0.98 (3)	C11—C12	1.391 (3)
N1—C7	1.297 (2)	C11—H11	0.9500

N1—C6	1.408 (2)	C4—C3	1.393 (3)
C18—C23	1.397 (3)	C4—H4	0.9500
C18—C19	1.401 (3)	C23—C22	1.389 (3)
C18—C17	1.465 (3)	C23—H23	0.9500
C17—H17	0.9500	C12—C13	1.392 (3)
C7—C8	1.505 (2)	C12—H12	0.9500
C6—C5	1.402 (3)	C15—C14	1.390 (3)
C6—C1	1.417 (2)	C15—H15	0.9500
C5—C4	1.384 (3)	C22—H22	0.9500
C5—H5	0.9500	C3—H3	0.9500
C10—C11	1.390 (3)	C19—C20	1.385 (3)
C10—C15	1.394 (3)	C19—H19	0.9500
C10—C9	1.517 (3)	C14—C13	1.387 (3)
C21—C20	1.385 (3)	C14—H14	0.9500
C21—C22	1.390 (3)	C13—H13	0.9500
C21—H21	0.9500	C20—H20	0.9500
C1—C2	1.396 (3)		

C17—C16—C7	125.22 (17)	C7—C8—H8B	109.3
C17—C16—H16	117.4	C9—C8—H8B	109.3
C7—C16—H16	117.4	H8A—C8—H8B	108.0
C1—N2—C9	121.80 (14)	C3—C2—C1	122.08 (17)
C1—N2—H	108.4 (16)	C3—C2—H2	119.0
C9—N2—H	109.6 (16)	C1—C2—H2	119.0
C7—N1—C6	120.14 (16)	C10—C11—C12	121.05 (19)
C23—C18—C19	117.82 (18)	C10—C11—H11	119.5
C23—C18—C17	119.03 (17)	C12—C11—H11	119.5
C19—C18—C17	123.15 (17)	C5—C4—C3	119.37 (18)
C16—C17—C18	125.63 (17)	C5—C4—H4	120.3
C16—C17—H17	117.2	C3—C4—H4	120.3
C18—C17—H17	117.2	C22—C23—C18	121.34 (18)
N1—C7—C16	116.24 (16)	C22—C23—H23	119.3
N1—C7—C8	121.51 (17)	C18—C23—H23	119.3
C16—C7—C8	122.23 (16)	C11—C12—C13	119.81 (19)
C5—C6—N1	117.42 (15)	C11—C12—H12	120.1
C5—C6—C1	118.90 (17)	C13—C12—H12	120.1
N1—C6—C1	123.25 (17)	C14—C15—C10	119.89 (18)
C4—C5—C6	121.84 (16)	C14—C15—H15	120.1
C4—C5—H5	119.1	C10—C15—H15	120.1
C6—C5—H5	119.1	C23—C22—C21	119.86 (19)
C11—C10—C15	118.97 (18)	C23—C22—H22	120.1
C11—C10—C9	117.83 (17)	C21—C22—H22	120.1
C15—C10—C9	123.11 (17)	C2—C3—C4	119.53 (18)
C20—C21—C22	119.6 (2)	C2—C3—H3	120.2
C20—C21—H21	120.2	C4—C3—H3	120.2
C22—C21—H21	120.2	C20—C19—C18	120.93 (19)
C2—C1—N2	120.20 (16)	C20—C19—H19	119.5
C2—C1—C6	118.27 (17)	C18—C19—H19	119.5

N2—C1—C6	121.12 (17)	C13—C14—C15	121.03 (19)
N2—C9—C10	111.69 (15)	C13—C14—H14	119.5
N2—C9—C8	109.02 (14)	C15—C14—H14	119.5
C10—C9—C8	110.54 (14)	C14—C13—C12	119.2 (2)
N2—C9—H9	108.5	C14—C13—H13	120.4
C10—C9—H9	108.5	C12—C13—H13	120.4
C8—C9—H9	108.5	C19—C20—C21	120.46 (19)
C7—C8—C9	111.64 (14)	C19—C20—H20	119.8
C7—C8—H8A	109.3	C21—C20—H20	119.8
C9—C8—H8A	109.3		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O01—H01···N1	0.92 (3)	2.01 (3)	2.9300 (19)	174 (3)
C11—H11···N1^{i}	0.95	2.51	3.426 (3)	161

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