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Crystal structure of 1-amino-2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile

Galal H. Elgemeie^a and Peter G. Jones^{b*}

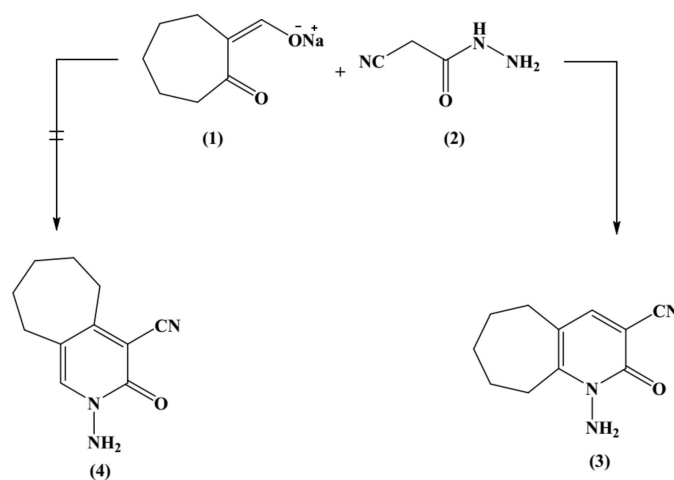
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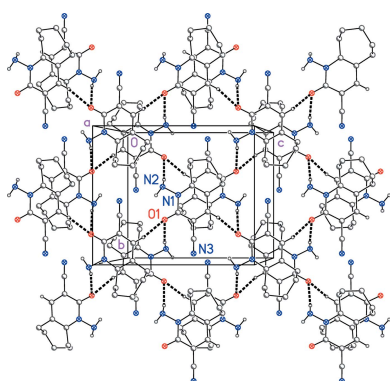
In the title compound, C₁₁H₁₃N₃O, the seven-membered ring adopts a conformation such that the three atoms not involved in the aromatic plane lie on the same side of that plane. One hydrazinic H atom forms an intramolecular hydrogen bond to the O atom; the other forms a classical intermolecular hydrogen bond to the O atom; the other forms a classical intermolecular hydrogen bond N—H···O, which combines with a 'weak' H_{ar}···O interaction to build up double layers of molecules parallel to the *bc* plane.

1. Chemical context

We have recently described various novel approaches for the synthesis of a new class of *N*-substituted amino derivatives of pyridines and pyrimidines (Elgemeie, Salah *et al.*, 2015; Elgemeie *et al.*, 2016). These compounds are important as pyrimidine ring systems that are not nucleoside analogs, and are interesting as antimetabolic agents (Elgemeie & Hamed, 2014; Elgemeie & Abd Elaziz, 2015). They have a greater selectivity for a broader range of human tumors, hence our interest in this class of compounds (Elgemeie, Abou-Zeid *et al.*, 2015; Elgemeie, Mohamed *et al.*, 2015).



We report here a novel one-step synthesis of a cycloheptane-ring-fused *N*-amino-2-pyridone derivative by reaction of the sodium salt of 2-(hydroxymethylene)-1-cycloheptanone (1) with a cyanoacetohydrazide (2). Thus, (1) reacted with (2) in piperidine acetate to give a product of molecular formula C₁₁H₁₃N₃O (*M*⁺ = 203), for which two isomeric structures, (3) and (4), seemed possible, corresponding to two possible modes of cyclization. Spectroscopic



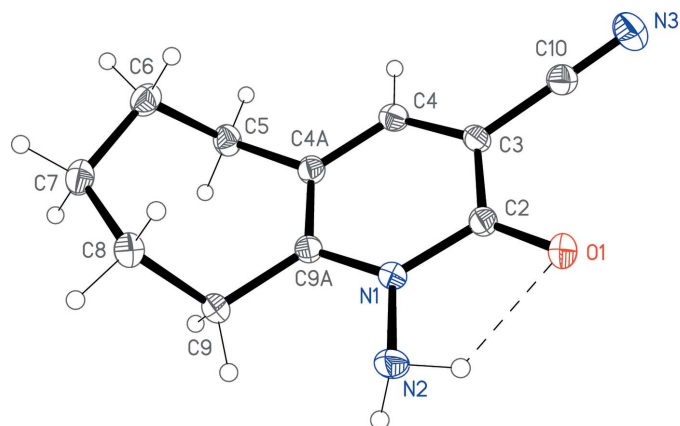


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond is shown as a dashed line (see Table 1)

data cannot differentiate between these structures, and therefore the crystal structure was determined, confirming the exclusive presence of tautomer (3) in the solid state. The formation of (3) from the reaction of (1) and (2) is assumed to

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N2—H01···O1	0.90 (2)	2.05 (2)	2.6255 (15)	120.4 (16)
N2—H02···O1 ⁱ	0.91 (2)	2.16 (2)	3.0225 (15)	158.2 (17)
C4—H4···O1 ⁱⁱ	0.95	2.45	3.2105 (16)	137
C9—H9A···O1 ⁱ	0.99	2.63	3.4903 (16)	146

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

proceed *via* initial addition of the active methylene carbon atom of (2) to the formyl group of (1) to give the favoured, kinetically controlled product (3). The ¹H NMR spectra of the product revealed the presence of an N—NH₂ group at $\delta = 6.4$ p.p.m. and a pyridine H-4 at 7.8 p.p.m. in solution.

2. Structural commentary

The structure of the title compound is shown in Fig. 1 and confirms the presence of tautomer (3) in the solid state. Molecular dimensions [*e.g.* the hydrazinic N1—N2 bond length of 1.4201 (15) Å] may be regarded as normal; an extensive structural investigation of alkyl-substituted 3-cyano-

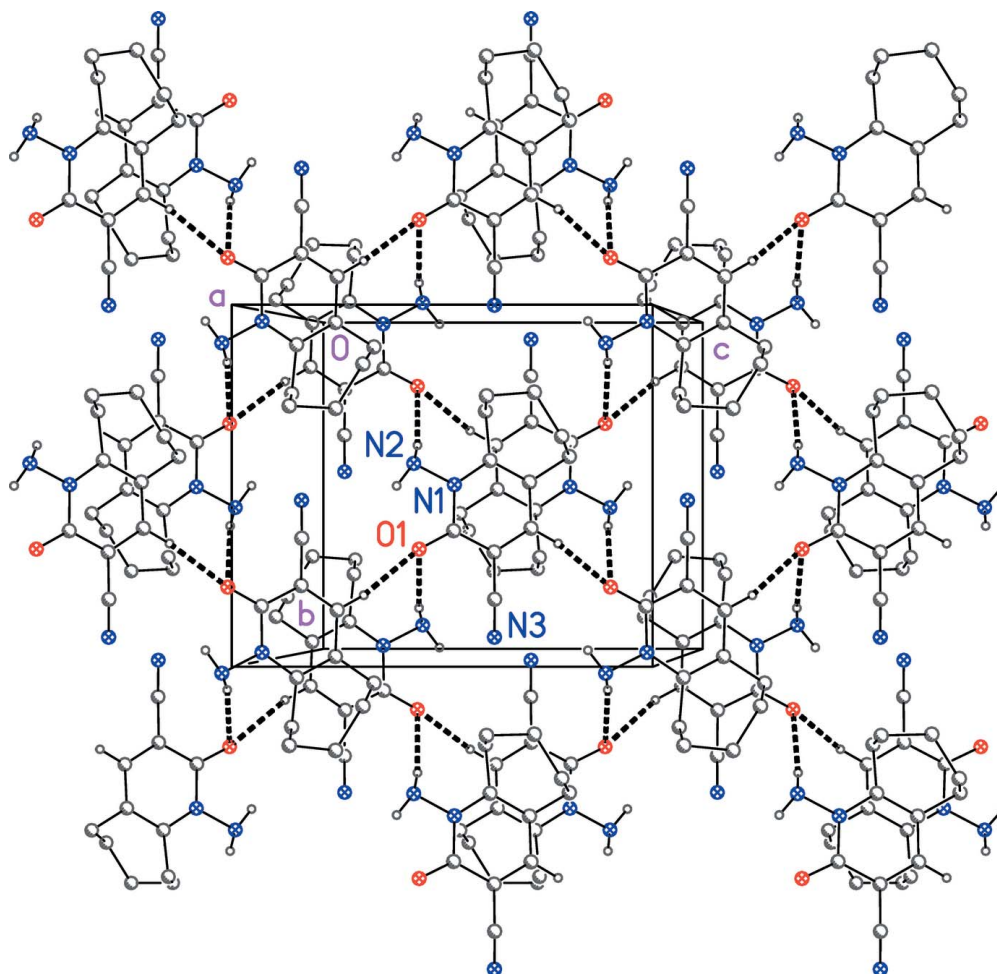


Figure 2
Crystal packing of the title compound, viewed approximately normal to the *bc* plane. Dashed lines indicate the hydrogen bonds (see Table 1), and for clarity H atoms not involved in hydrogen bonding have been omitted.

2-pyridones (with an unsubstituted NH function in the ring) was published by Fischer *et al.* (2004). The seven-membered ring adopts a conformation such that all three atoms C6, C7 and C8 lie to the same side of the plane formed by the pyridone ring together with C5 and C9; the respective deviations from this plane are 1.480 (2), 1.616 (3) and 1.470 (2) Å.

3. Supramolecular features

The classical hydrogen-bond donor N1—H01 is only involved in intramolecular hydrogen bonding (Fig. 1 and Table 1). The second such donor N1—H02 forms a classical hydrogen bond to the acceptor O1 of a neighbouring molecule related by the 2₁ screw axis. Additionally, the 'weak' but quite short hydrogen bond C4—H4...O1 links molecules related by the *c* glide plane. The overall effect is to build up double layers of molecules (Fig. 2 and Table 1) parallel to the *bc* plane, in which the top and bottom molecules of the layer are related by inversion.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.37, last update May 2016; Groom *et al.*, 2016) revealed four other examples of the cyclohepta[*b*]pyridin-2-one ring system: refcodes AHEQAF (Elgemeie *et al.*, 2002), ATUYAP and IBATUB (Albov *et al.*, 2004*a,b*) and QAHLOB (Fischer *et al.*, 2004).

5. Synthesis and crystallization

A solution of the sodium salt of 2-(hydroxymethylene)-1-cycloheptanone [(1); 1.60 g, 0.01 mol], *N*-cyanoaceto-hydrazide [(2); 0.09 g, 0.01 mol] and piperidine acetate (1 ml) in water (30 ml) and ethanol (30 ml) was refluxed for 10 min. Acetic acid (1.5 ml) was added to the hot solution. The precipitated solid was collected by filtration and crystallized from ethanol, giving colourless plate-like crystals (yield 85%, m.p. 508 K).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The NH hydrogens were located in a difference Fourier map and freely refined. The C-bound H atoms were included using a riding model starting from calculated positions: C—H = 0.95–0.99 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

References

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Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₁ H ₁₃ N ₃ O
<i>M_r</i>	203.24
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.5680 (4), 10.0475 (4), 11.6778 (5)
β (°)	103.272 (4)
<i>V</i> (Å ³)	978.46 (7)
<i>Z</i>	4
Radiation type	Cu Kα
μ (mm ⁻¹)	0.74
Crystal size (mm)	0.10 × 0.10 × 0.05
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Atlas Nova
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)
<i>T_{min}</i> , <i>T_{max}</i>	0.795, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	19833, 2046, 1674
<i>R_{int}</i>	0.062
(sin θ/λ) _{max} (Å ⁻¹)	0.630
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.039, 0.109, 1.06
No. of reflections	2046
No. of parameters	144
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.28, -0.30

Computer programs: (*CrysAlis PRO*; Agilent, 2010), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *XP* (Siemens, 1994).

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supporting information

Acta Cryst. (2016). E72, 1239-1241 [doi:10.1107/S2056989016012196]

Crystal structure of 1-amino-2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile

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Computing details

Data collection: (*CrysAlis PRO*; Agilent, 2010); cell refinement: (*CrysAlis PRO*; Agilent, 2010); data reduction: (*CrysAlis PRO*; Agilent, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

1-Amino-2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile

Crystal data

$C_{11}H_{13}N_3O$

$M_r = 203.24$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.5680$ (4) Å

$b = 10.0475$ (4) Å

$c = 11.6778$ (5) Å

$\beta = 103.272$ (4)°

$V = 978.46$ (7) Å³

$Z = 4$

$F(000) = 432$

$D_x = 1.380$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 6583 reflections

$\theta = 3.9$ – 76.1 °

$\mu = 0.74$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.10 \times 0.10 \times 0.05$ mm

Data collection

Oxford Diffraction Xcalibur Atlas Nova diffractometer

Radiation source: sealed X-ray tube, Nova (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.3543 pixels mm⁻¹

ω -scan

Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)

$T_{\min} = 0.795$, $T_{\max} = 1.000$

19833 measured reflections

2046 independent reflections

1674 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\max} = 76.1$ °, $\theta_{\min} = 5.3$ °

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.109$

$S = 1.06$

2046 reflections

144 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.2053P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

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.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
 $8.5275 (0.0007) x + 0.7466 (0.0052) y - 3.3783 (0.0058) z = 0.3993 (0.0040)$

* $-0.0117 (0.0008) N1 * 0.0031 (0.0009) C2 * 0.0072 (0.0009) C3 * -0.0090 (0.0009) C4 * 0.0007 (0.0009) C4A * 0.0097 (0.0009) C9A$
 $1.4803 (0.0024) C6 1.6155 (0.0026) C7 1.4700 (0.0023) C8$

Rms deviation of fitted atoms = 0.0079

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 $F^2 > 2\sigma(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.14967 (13)	0.49677 (10)	0.37289 (9)	0.0161 (3)
C2	0.13750 (15)	0.63589 (13)	0.36852 (11)	0.0168 (3)
C3	0.17684 (15)	0.69947 (13)	0.48064 (12)	0.0168 (3)
C4	0.22210 (15)	0.62730 (13)	0.58375 (11)	0.0173 (3)
H4	0.2451	0.6727	0.6571	0.021*
C4A	0.23457 (16)	0.48899 (13)	0.58178 (11)	0.0169 (3)
C5	0.29860 (17)	0.41294 (13)	0.69430 (11)	0.0197 (3)
H5A	0.2243	0.3391	0.7000	0.024*
H5B	0.3025	0.4729	0.7622	0.024*
C6	0.46697 (17)	0.35590 (14)	0.70100 (12)	0.0218 (3)
H6A	0.5315	0.4231	0.6703	0.026*
H6B	0.5197	0.3395	0.7845	0.026*
C7	0.46682 (17)	0.22636 (14)	0.63197 (12)	0.0212 (3)
H7A	0.5787	0.1942	0.6447	0.025*
H7B	0.4055	0.1587	0.6651	0.025*
C8	0.39649 (16)	0.23537 (13)	0.49953 (12)	0.0199 (3)
H8A	0.4085	0.1478	0.4637	0.024*
H8B	0.4596	0.3008	0.4656	0.024*
C9	0.21817 (16)	0.27618 (13)	0.46552 (12)	0.0186 (3)
H9A	0.1708	0.2467	0.3841	0.022*
H9B	0.1597	0.2312	0.5185	0.022*
C9A	0.19855 (15)	0.42414 (13)	0.47385 (11)	0.0159 (3)
C10	0.17377 (16)	0.84204 (14)	0.48160 (12)	0.0200 (3)
N2	0.12201 (15)	0.43233 (12)	0.26182 (10)	0.0207 (3)
H01	0.090 (2)	0.500 (2)	0.2110 (18)	0.036 (5)*
H02	0.037 (2)	0.376 (2)	0.2551 (16)	0.030 (5)*
N3	0.17469 (17)	0.95620 (12)	0.48127 (12)	0.0298 (3)
O1	0.09637 (12)	0.69230 (9)	0.27097 (8)	0.0208 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0174 (6)	0.0137 (5)	0.0162 (5)	-0.0004 (4)	0.0016 (4)	-0.0012 (4)
C2	0.0134 (6)	0.0151 (6)	0.0214 (7)	0.0001 (4)	0.0028 (5)	0.0014 (5)
C3	0.0150 (6)	0.0129 (6)	0.0223 (6)	-0.0009 (4)	0.0037 (5)	-0.0012 (5)
C4	0.0154 (6)	0.0171 (6)	0.0199 (6)	-0.0005 (5)	0.0047 (5)	-0.0024 (5)
C4A	0.0153 (6)	0.0162 (6)	0.0192 (6)	0.0000 (5)	0.0036 (5)	0.0012 (5)
C5	0.0240 (7)	0.0182 (6)	0.0170 (6)	0.0015 (5)	0.0051 (5)	0.0018 (5)
C6	0.0212 (7)	0.0241 (7)	0.0182 (6)	0.0018 (5)	0.0006 (5)	0.0020 (5)
C7	0.0197 (7)	0.0204 (7)	0.0229 (7)	0.0036 (5)	0.0037 (5)	0.0044 (5)
C8	0.0199 (7)	0.0182 (6)	0.0215 (6)	0.0026 (5)	0.0048 (5)	0.0005 (5)
C9	0.0193 (7)	0.0137 (6)	0.0218 (6)	-0.0002 (5)	0.0026 (5)	-0.0003 (5)
C9A	0.0133 (6)	0.0145 (6)	0.0195 (6)	-0.0006 (4)	0.0030 (5)	0.0008 (4)
C10	0.0183 (6)	0.0182 (7)	0.0227 (6)	0.0002 (5)	0.0033 (5)	-0.0007 (5)
N2	0.0272 (7)	0.0176 (6)	0.0154 (5)	-0.0018 (5)	0.0006 (5)	-0.0033 (4)
N3	0.0340 (8)	0.0182 (6)	0.0366 (7)	0.0000 (5)	0.0068 (6)	-0.0004 (5)
O1	0.0237 (5)	0.0170 (4)	0.0199 (5)	0.0012 (4)	0.0012 (4)	0.0039 (4)

Geometric parameters (Å, °)

N1—C9A	1.3680 (16)	C10—N3	1.1471 (19)
N1—C2	1.4017 (16)	C4—H4	0.9500
N1—N2	1.4201 (15)	C5—H5A	0.9900
C2—O1	1.2486 (16)	C5—H5B	0.9900
C2—C3	1.4260 (18)	C6—H6A	0.9900
C3—C4	1.3826 (18)	C6—H6B	0.9900
C3—C10	1.4328 (18)	C7—H7A	0.9900
C4—C4A	1.3943 (18)	C7—H7B	0.9900
C4A—C9A	1.3892 (18)	C8—H8A	0.9900
C4A—C5	1.5101 (17)	C8—H8B	0.9900
C5—C6	1.5375 (19)	C9—H9A	0.9900
C6—C7	1.5308 (19)	C9—H9B	0.9900
C7—C8	1.5283 (18)	N2—H01	0.90 (2)
C8—C9	1.5432 (18)	N2—H02	0.91 (2)
C9—C9A	1.5018 (17)		
C9A—N1—C2	124.66 (11)	C4A—C5—H5B	109.1
C9A—N1—N2	119.86 (10)	C6—C5—H5B	109.1
C2—N1—N2	115.22 (10)	H5A—C5—H5B	107.8
O1—C2—N1	119.27 (11)	C7—C6—H6A	108.8
O1—C2—C3	126.31 (12)	C5—C6—H6A	108.8
N1—C2—C3	114.42 (11)	C7—C6—H6B	108.8
C4—C3—C2	121.64 (12)	C5—C6—H6B	108.8
C4—C3—C10	121.30 (12)	H6A—C6—H6B	107.7
C2—C3—C10	117.01 (12)	C8—C7—H7A	108.3
C3—C4—C4A	121.04 (12)	C6—C7—H7A	108.3
C9A—C4A—C4	118.72 (12)	C8—C7—H7B	108.3

C9A—C4A—C5	120.85 (12)	C6—C7—H7B	108.3
C4—C4A—C5	120.26 (12)	H7A—C7—H7B	107.4
C4A—C5—C6	112.46 (11)	C7—C8—H8A	108.7
C7—C6—C5	113.75 (11)	C9—C8—H8A	108.7
C8—C7—C6	115.79 (11)	C7—C8—H8B	108.7
C7—C8—C9	114.41 (11)	C9—C8—H8B	108.7
C9A—C9—C8	111.42 (11)	H8A—C8—H8B	107.6
N1—C9A—C4A	119.48 (12)	C9A—C9—H9A	109.3
N1—C9A—C9	119.26 (11)	C8—C9—H9A	109.3
C4A—C9A—C9	121.22 (12)	C9A—C9—H9B	109.3
N3—C10—C3	178.28 (16)	C8—C9—H9B	109.3
C3—C4—H4	119.5	H9A—C9—H9B	108.0
C4A—C4—H4	119.5	N1—N2—H01	102.6 (13)
C4A—C5—H5A	109.1	N1—N2—H02	108.8 (12)
C6—C5—H5A	109.1	H01—N2—H02	107.1 (17)
C9A—N1—C2—O1	177.72 (12)	C4A—C5—C6—C7	-80.60 (15)
N2—N1—C2—O1	3.59 (17)	C5—C6—C7—C8	61.27 (16)
C9A—N1—C2—C3	-1.60 (18)	C6—C7—C8—C9	-61.68 (16)
N2—N1—C2—C3	-175.73 (11)	C7—C8—C9—C9A	80.91 (14)
O1—C2—C3—C4	-179.57 (13)	C2—N1—C9A—C4A	2.30 (19)
N1—C2—C3—C4	-0.30 (18)	N2—N1—C9A—C4A	176.17 (12)
O1—C2—C3—C10	-2.1 (2)	C2—N1—C9A—C9	-175.43 (12)
N1—C2—C3—C10	177.22 (11)	N2—N1—C9A—C9	-1.55 (18)
C2—C3—C4—C4A	1.5 (2)	C4—C4A—C9A—N1	-0.99 (18)
C10—C3—C4—C4A	-175.93 (13)	C5—C4A—C9A—N1	-176.19 (12)
C3—C4—C4A—C9A	-0.82 (19)	C4—C4A—C9A—C9	176.68 (12)
C3—C4—C4A—C5	174.41 (12)	C5—C4A—C9A—C9	1.48 (19)
C9A—C4A—C5—C6	66.67 (16)	C8—C9—C9A—N1	109.73 (13)
C4—C4A—C5—C6	-108.45 (14)	C8—C9—C9A—C4A	-67.96 (16)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H01 \cdots O1	0.90 (2)	2.05 (2)	2.6255 (15)	120.4 (16)
N2—H02 \cdots O1 ⁱ	0.91 (2)	2.16 (2)	3.0225 (15)	158.2 (17)
C4—H4 \cdots O1 ⁱⁱ	0.95	2.45	3.2105 (16)	137
C9—H9A \cdots O1 ⁱ	0.99	2.63	3.4903 (16)	146

Symmetry codes: (i) $-x, y-1/2, -z+1/2$; (ii) $x, -y+3/2, z+1/2$.