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N′-Cyclododecylidenepyridine-4-carbohydrazide

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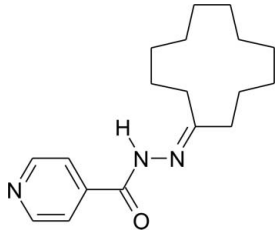
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.111; data-to-parameter ratio = 14.4.

The title compound, $C_{18}H_{27}N_3O$, is a derivative of the antituberculosis drug isoniazid (systematic name: pyridine-4-carbohydrazidei). The crystal structure consists of repeating $C(4)$ chains along the b axis, formed by N—H···O hydrogen bonds with adjacent amide functional groups that are related by a b-glide plane. The cyclododecyl ring has the same approximately 'square' conformation, as seen in the parent hydrocarbon cyclododecane.

Related literature

For hydrogen-bonding motifs, see: Bernstein *et al.* (1995). For cycloalkane ring conformations, see: Dale (1966).



Experimental

Crystal data

$C_{18}H_{27}N_3O$
M_r = 301.43
Orthorhombic, *Pbca*
a = 14.8450 (6) Å
b = 8.0980 (4) Å
c = 27.3910 (11) Å
V = 3292.8 (2) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 100 K
0.44 × 0.34 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
T_{min} = 0.779, T_{max} = 0.890
23848 measured reflections
2931 independent reflections
2535 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.08
2931 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88 (2)	2.15 (2)	3.0122 (15)	164.7 (16)

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2532).

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supporting information

Acta Cryst. (2012). E**68**, o1205 [https://doi.org/10.1107/S1600536812011774]

N′-Cyclododecylidenepyridine-4-carbohydrazide

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S1. Comment

Fig. 1 shows the atomic numbering scheme of the title compound. The amide functional groups form a torsion angle of 38.54 (17)° with the pyridine ring. Fig. 2 shows the *C*(4) (Bernstein *et al.*, 1995) hydrogen bonded ring formed with adjacent amide functional groups, leading to a chain along the *b*-axis. The cyclododecyl ring has a square conformation, as seen in the related cycloalkane $C_{12}H_{24}$ ring (Dale, 1966).

S2. Experimental

A stoichiometric amount in the ratio of 1:1 of isonicotinic acid hydrazide to cyclododecanone was dissolved in 5 ml of methanol. The solution was refluxed for a few hours, and left to cool to room temperature. Colourless, block-like crystals were harvested after slow evaporation over a few days at ambient conditions.

S3. Refinement

The C-bound H atoms were geometrically placed (C—H bond lengths of 0.95 (aromatic CH) and 0.99 (methylene CH_2) Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The N-bound H atoms were located in the difference map and coordinates refined freely together with their isotropic thermal parameters.

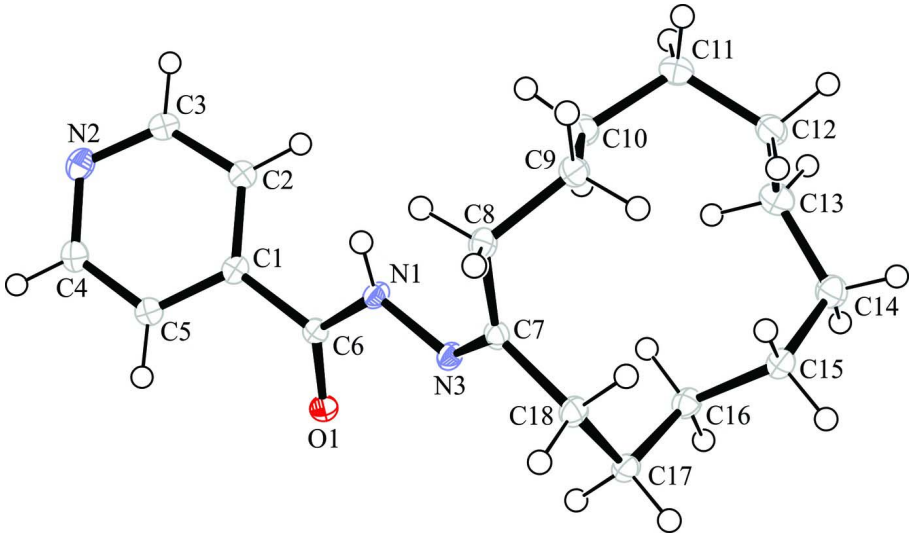

Figure 1

The asymmetric unit of (I) showing the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

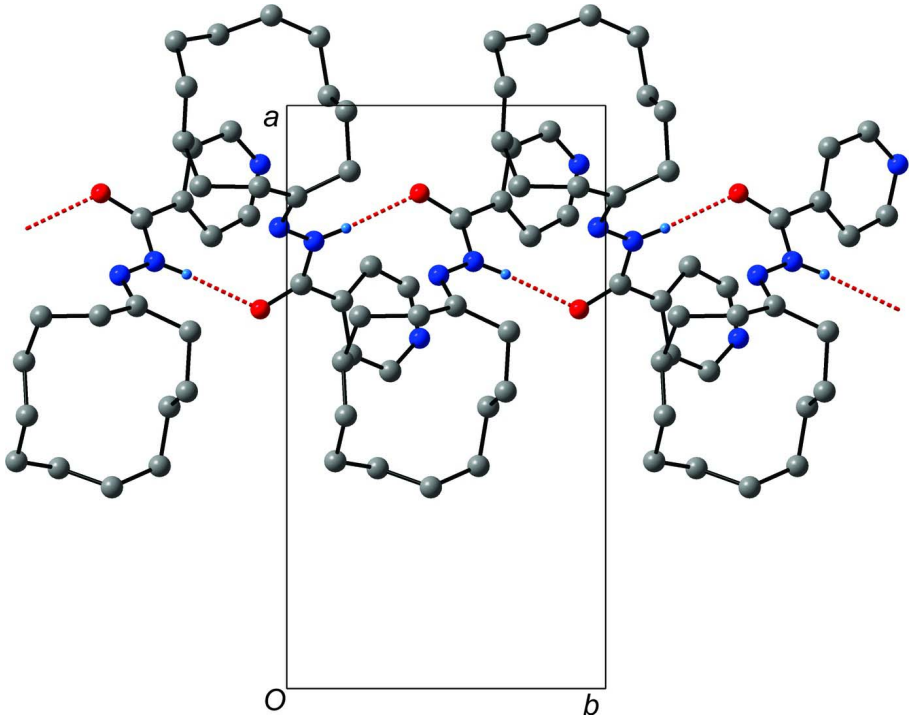

Figure 2

Hydrogen bonding chain showing the C(4) hydrogen bonded chains. Intermolecular N—H···O hydrogen bonds are shown as dashed red lines.

N′-Cyclododecylidenepyridine-4-carbohydrazide

Crystal data

$C_{18}H_{27}N_3O$
$M_r = 301.43$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 14.8450\ (6)$ Å
$b = 8.0980\ (4)$ Å
$c = 27.3910\ (11)$ Å
$V = 3292.8\ (2)$ Å^3
$Z = 8$
$F(000) = 1312$
$D_x = 1.216$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 9464 reflections
$\theta = 3.0$–$67.5°$
$\mu = 0.60$ mm^{-1}
$T = 100$ K
Block, colourless
0.44 × 0.34 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini ultra diffractometer
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
$T_{min} = 0.779$, $T_{max} = 0.890$
23848 measured reflections
2931 independent reflections
2535 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 67.0°$, $\theta_{min} = 3.2°$
$h = -17 \rightarrow 16$
$k = -9 \rightarrow 9$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.08$
2931 reflections
203 parameters
0 restraints

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.6785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Experimental. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm in CrysAlisPro.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.83538 (9)	0.67344 (15)	0.71252 (5)	0.0140 (3)
C2	0.77610 (9)	0.76355 (16)	0.74142 (5)	0.0151 (3)
H2	0.713	0.7447	0.7397	0.018*
C3	0.81105 (9)	0.88164 (16)	0.77286 (5)	0.0171 (3)
H3	0.7701	0.9417	0.7927	0.02*
C4	0.95549 (9)	0.82425 (17)	0.74991 (5)	0.0185 (3)
H4	1.0183	0.8447	0.7527	0.022*
C5	0.92753 (9)	0.70136 (16)	0.71813 (5)	0.0157 (3)
H5	0.9702	0.6374	0.7005	0.019*
C6	0.80631 (9)	0.54546 (16)	0.67606 (4)	0.0131 (3)
C7	0.65632 (8)	0.52936 (16)	0.57794 (5)	0.0144 (3)
C8	0.61244 (9)	0.69684 (16)	0.57370 (5)	0.0160 (3)
H8A	0.6251	0.7436	0.541	0.019*
H8B	0.6386	0.7721	0.5984	0.019*
C9	0.50972 (9)	0.68614 (17)	0.58126 (5)	0.0179 (3)
H9A	0.4828	0.7957	0.5746	0.022*
H9B	0.4843	0.607	0.5574	0.022*
C10	0.48338 (9)	0.63169 (18)	0.63275 (5)	0.0208 (3)
H10A	0.5026	0.7177	0.6562	0.025*
H10B	0.5163	0.5289	0.6408	0.025*
C11	0.38197 (9)	0.60107 (18)	0.63905 (5)	0.0221 (3)
H11A	0.3691	0.5856	0.6742	0.027*
H11B	0.349	0.7008	0.6281	0.027*
C12	0.34546 (9)	0.45158 (17)	0.61105 (5)	0.0202 (3)
H12A	0.2788	0.4539	0.6123	0.024*
H12B	0.3636	0.4613	0.5764	0.024*
C13	0.37813 (10)	0.28492 (17)	0.63069 (5)	0.0219 (3)
H13A	0.4364	0.3016	0.6475	0.026*
H13B	0.3343	0.2447	0.6552	0.026*

C14	0.39013 (10)	0.15214 (17)	0.59157 (6)	0.0224 (3)
H14A	0.3336	0.1432	0.5726	0.027*
H14B	0.4004	0.0446	0.6078	0.027*
C15	0.46814 (9)	0.18511 (17)	0.55621 (5)	0.0190 (3)
H15A	0.4653	0.1034	0.5294	0.023*
H15B	0.4601	0.2961	0.5417	0.023*
C16	0.56119 (9)	0.17631 (16)	0.57975 (5)	0.0178 (3)
H16A	0.5722	0.0615	0.5906	0.021*
H16B	0.5615	0.2476	0.6091	0.021*
C17	0.63832 (9)	0.22928 (17)	0.54617 (5)	0.0178 (3)
H17A	0.6962	0.1979	0.5615	0.021*
H17B	0.6333	0.1683	0.515	0.021*
C18	0.63976 (9)	0.41420 (17)	0.53531 (5)	0.0168 (3)
H18A	0.687	0.4349	0.5105	0.02*
H18B	0.5813	0.4443	0.5204	0.02*
N1	0.73263 (7)	0.58502 (14)	0.64919 (4)	0.0148 (3)
H1	0.7106 (12)	0.686 (2)	0.6498 (6)	0.024 (4)*
N2	0.89901 (8)	0.91633 (14)	0.77697 (4)	0.0191 (3)
N3	0.70898 (7)	0.47642 (14)	0.61174 (4)	0.0148 (3)
O1	0.84951 (6)	0.41677 (11)	0.67137 (3)	0.0159 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0179 (6)	0.0130 (6)	0.0113 (6)	0.0000 (5)	−0.0025 (5)	0.0028 (5)
C2	0.0164 (6)	0.0153 (6)	0.0136 (6)	0.0009 (5)	−0.0012 (5)	0.0028 (5)
C3	0.0202 (7)	0.0163 (6)	0.0147 (6)	0.0022 (5)	−0.0002 (5)	0.0004 (5)
C4	0.0180 (6)	0.0203 (7)	0.0171 (6)	−0.0029 (5)	−0.0012 (5)	0.0013 (5)
C5	0.0174 (7)	0.0160 (6)	0.0136 (6)	−0.0001 (5)	0.0007 (5)	0.0018 (5)
C6	0.0146 (6)	0.0135 (6)	0.0113 (6)	−0.0011 (5)	0.0014 (5)	0.0015 (5)
C7	0.0128 (6)	0.0164 (7)	0.0141 (6)	−0.0024 (5)	0.0012 (5)	−0.0004 (5)
C8	0.0169 (7)	0.0164 (6)	0.0146 (6)	−0.0010 (5)	−0.0031 (5)	0.0005 (5)
C9	0.0166 (7)	0.0182 (7)	0.0190 (7)	0.0013 (5)	−0.0023 (5)	−0.0009 (5)
C10	0.0189 (7)	0.0257 (7)	0.0178 (7)	−0.0006 (6)	0.0011 (5)	−0.0046 (6)
C11	0.0189 (7)	0.0247 (7)	0.0227 (7)	0.0018 (6)	0.0041 (6)	−0.0035 (6)
C12	0.0160 (6)	0.0228 (7)	0.0220 (7)	0.0016 (5)	0.0001 (5)	−0.0004 (6)
C13	0.0215 (7)	0.0240 (7)	0.0201 (7)	0.0010 (6)	0.0031 (6)	0.0033 (6)
C14	0.0208 (7)	0.0186 (7)	0.0278 (8)	−0.0017 (5)	0.0007 (6)	0.0002 (6)
C15	0.0200 (7)	0.0179 (7)	0.0192 (7)	−0.0008 (5)	−0.0015 (5)	−0.0034 (5)
C16	0.0203 (7)	0.0141 (6)	0.0192 (7)	0.0007 (5)	−0.0027 (5)	0.0002 (5)
C17	0.0174 (6)	0.0182 (7)	0.0177 (7)	0.0016 (5)	−0.0028 (5)	−0.0048 (5)
C18	0.0172 (6)	0.0197 (7)	0.0134 (6)	−0.0012 (5)	−0.0008 (5)	−0.0018 (5)
N1	0.0168 (5)	0.0131 (6)	0.0145 (5)	0.0014 (4)	−0.0039 (4)	−0.0029 (4)
N2	0.0234 (6)	0.0178 (6)	0.0161 (6)	−0.0027 (5)	−0.0025 (5)	−0.0003 (4)
N3	0.0159 (5)	0.0157 (5)	0.0127 (5)	−0.0020 (4)	−0.0007 (4)	−0.0027 (4)
O1	0.0165 (5)	0.0145 (5)	0.0166 (5)	0.0011 (3)	0.0001 (4)	−0.0001 (4)

Geometric parameters (Å, º)

C1—C2	1.3905 (19)	C11—C12	1.532 (2)
C1—C5	1.3950 (18)	C11—H11A	0.99
C1—C6	1.5026 (17)	C11—H11B	0.99
C2—C3	1.3875 (19)	C12—C13	1.5317 (19)
C2—H2	0.95	C12—H12A	0.99
C3—N2	1.3404 (18)	C12—H12B	0.99
C3—H3	0.95	C13—C14	1.529 (2)
C4—N2	1.3447 (19)	C13—H13A	0.99
C4—C5	1.3857 (19)	C13—H13B	0.99
C4—H4	0.95	C14—C15	1.533 (2)
C5—H5	0.95	C14—H14A	0.99
C6—O1	1.2304 (16)	C14—H14B	0.99
C6—N1	1.3567 (17)	C15—C16	1.5262 (18)
C7—N3	1.2853 (17)	C15—H15A	0.99
C7—C8	1.5091 (18)	C15—H15B	0.99
C7—C18	1.5144 (18)	C16—C17	1.5301 (19)
C8—C9	1.5414 (18)	C16—H16A	0.99
C8—H8A	0.99	C16—H16B	0.99
C8—H8B	0.99	C17—C18	1.5268 (19)
C9—C10	1.5286 (19)	C17—H17A	0.99
C9—H9A	0.99	C17—H17B	0.99
C9—H9B	0.99	C18—H18A	0.99
C10—C11	1.5354 (19)	C18—H18B	0.99
C10—H10A	0.99	N1—N3	1.3961 (15)
C10—H10B	0.99	N1—H1	0.88 (2)
C2—C1—C5	118.21 (12)	C13—C12—H12A	108.7
C2—C1—C6	123.96 (12)	C11—C12—H12A	108.7
C5—C1—C6	117.82 (12)	C13—C12—H12B	108.7
C3—C2—C1	118.59 (12)	C11—C12—H12B	108.7
C3—C2—H2	120.7	H12A—C12—H12B	107.6
C1—C2—H2	120.7	C14—C13—C12	114.23 (12)
N2—C3—C2	124.11 (12)	C14—C13—H13A	108.7
N2—C3—H3	117.9	C12—C13—H13A	108.7
C2—C3—H3	117.9	C14—C13—H13B	108.7
N2—C4—C5	123.89 (13)	C12—C13—H13B	108.7
N2—C4—H4	118.1	H13A—C13—H13B	107.6
C5—C4—H4	118.1	C13—C14—C15	114.11 (11)
C4—C5—C1	118.65 (12)	C13—C14—H14A	108.7
C4—C5—H5	120.7	C15—C14—H14A	108.7
C1—C5—H5	120.7	C13—C14—H14B	108.7
O1—C6—N1	124.31 (12)	C15—C14—H14B	108.7
O1—C6—C1	120.26 (11)	H14A—C14—H14B	107.6
N1—C6—C1	115.42 (11)	C16—C15—C14	114.11 (12)
N3—C7—C8	128.15 (12)	C16—C15—H15A	108.7
N3—C7—C18	116.66 (12)	C14—C15—H15A	108.7

C8—C7—C18	115.09 (11)	C16—C15—H15B	108.7
C7—C8—C9	111.48 (11)	C14—C15—H15B	108.7
C7—C8—H8A	109.3	H15A—C15—H15B	107.6
C9—C8—H8A	109.3	C15—C16—C17	114.22 (11)
C7—C8—H8B	109.3	C15—C16—H16A	108.7
C9—C8—H8B	109.3	C17—C16—H16A	108.7
H8A—C8—H8B	108	C15—C16—H16B	108.7
C10—C9—C8	113.16 (11)	C17—C16—H16B	108.7
C10—C9—H9A	108.9	H16A—C16—H16B	107.6
C8—C9—H9A	108.9	C18—C17—C16	113.74 (11)
C10—C9—H9B	108.9	C18—C17—H17A	108.8
C8—C9—H9B	108.9	C16—C17—H17A	108.8
H9A—C9—H9B	107.8	C18—C17—H17B	108.8
C9—C10—C11	113.64 (12)	C16—C17—H17B	108.8
C9—C10—H10A	108.8	H17A—C17—H17B	107.7
C11—C10—H10A	108.8	C7—C18—C17	117.13 (11)
C9—C10—H10B	108.8	C7—C18—H18A	108
C11—C10—H10B	108.8	C17—C18—H18A	108
H10A—C10—H10B	107.7	C7—C18—H18B	108
C12—C11—C10	114.72 (11)	C17—C18—H18B	108
C12—C11—H11A	108.6	H18A—C18—H18B	107.3
C10—C11—H11A	108.6	C6—N1—N3	116.91 (11)
C12—C11—H11B	108.6	C6—N1—H1	120.5 (12)
C10—C11—H11B	108.6	N3—N1—H1	120.4 (12)
H11A—C11—H11B	107.6	C3—N2—C4	116.42 (12)
C13—C12—C11	114.10 (12)	C7—N3—N1	118.17 (11)

C5—C1—C2—C3	2.74 (18)	C11—C12—C13—C14	−146.98 (12)
C6—C1—C2—C3	−178.29 (12)	C12—C13—C14—C15	68.58 (16)
C1—C2—C3—N2	0.7 (2)	C13—C14—C15—C16	67.05 (16)
N2—C4—C5—C1	1.9 (2)	C14—C15—C16—C17	−173.04 (11)
C2—C1—C5—C4	−3.94 (18)	C15—C16—C17—C18	70.36 (15)
C6—C1—C5—C4	177.03 (11)	N3—C7—C18—C17	33.46 (17)
C2—C1—C6—O1	−140.43 (13)	C8—C7—C18—C17	−149.87 (11)
C5—C1—C6—O1	38.54 (17)	C16—C17—C18—C7	64.33 (15)
C2—C1—C6—N1	41.05 (17)	O1—C6—N1—N3	−4.55 (19)
C5—C1—C6—N1	−139.98 (12)	C1—C6—N1—N3	173.91 (10)
N3—C7—C8—C9	−110.81 (15)	C2—C3—N2—C4	−2.77 (19)
C18—C7—C8—C9	72.97 (14)	C5—C4—N2—C3	1.44 (19)
C7—C8—C9—C10	64.83 (15)	C8—C7—N3—N1	−2.22 (19)
C8—C9—C10—C11	−173.28 (11)	C18—C7—N3—N1	173.95 (11)
C9—C10—C11—C12	68.04 (16)	C6—N1—N3—C7	−161.67 (12)
C10—C11—C12—C13	68.83 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1···O1^{i}	0.88 (2)	2.15 (2)	3.0122 (15)	164.7 (16)

Symmetry code: (i) −*x*+3/2, *y*+1/2, *z*.