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(E)-2-Cyano-N'-(1,2,3,4-tetrahydronaphthalen-1-ylidene)acetohydrazideMohamed A. Al-Omar,^{a,b} Nagy M. Khalifa,^{b,c} Hazem A. Ghabbour,^a Tze Shyang Chia^d and Hoong-Kun Fun^{d,*†}

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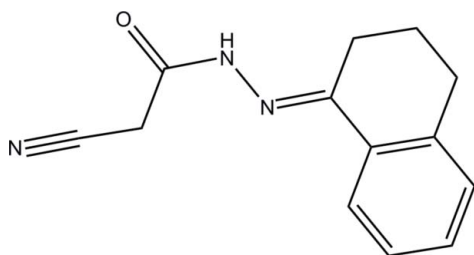
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.120; data-to-parameter ratio = 11.9.

In the title compound, $\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}$, the tetrahydrobenzene ring adopts a half-boat (envelope) conformation. The mean plane of the tetrahydronaphthalene ring system forms a dihedral angle of $9.56(6)^\circ$ with the mean plane of the cyanoacetohydrazide group. In the crystal, inversion dimers linked by pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds generate $R_2^2(8)$ loops. The dimers are connected by $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds into a chain propagating along $[101]$. The crystal packing also features $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For background to tetralin, see: Dutta *et al.* (2002); Taddei *et al.* (2002); Zaghary *et al.* (2005); Bahgat & Khalifa (2006); El Nezhawy *et al.* (2009); Khalifa *et al.* (2008). For ring puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}$
 $M_r = 227.26$
Triclinic, $P\bar{1}$

$a = 7.6414(1)$ Å
 $b = 7.6748(1)$ Å
 $c = 10.5644(2)$ Å

$\alpha = 109.589(1)^\circ$
 $\beta = 91.405(1)^\circ$
 $\gamma = 93.260(1)^\circ$
 $V = 582.13(2)$ Å³
 $Z = 2$

Cu $K\alpha$ radiation
 $\mu = 0.69$ mm⁻¹
 $T = 296$ K
 $0.59 \times 0.51 \times 0.40$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.687$, $T_{\max} = 0.771$

5749 measured reflections
1898 independent reflections
1762 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.120$
 $S = 1.05$
1898 reflections
159 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C5–C10 ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O1}^{\text{i}}$	0.91 (2)	1.96 (2)	2.8640 (17)	174.7 (19)
$\text{C10}-\text{H10A}\cdots\text{N3}^{\text{ii}}$	0.93	2.58	3.491 (3)	167
$\text{C2}-\text{H2A}\cdots\text{Cg1}^{\text{iii}}$	0.97	2.80	3.6775 (17)	152

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6781).

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supplementary materials

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(E)-2-Cyano-N'-(1,2,3,4-tetrahydronaphthalen-1-ylidene)acetohydrazide

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Comment

Tetralins (tetrahydronaphthalene derivatives) are of increasing interest since many of these compounds play a vital role in the biological activities because of their biological potentialities, for example, as potent agonists for D2-type receptors (Dutta *et al.*, 2002), a treatment of Alzheimer's disease (Taddei *et al.*, 2002) and as anti-cancer agents (Zaghary *et al.*, 2005). Also, we found that certain substituted tetralin and heterocyclic derivatives show inhibition for cercarial serine protease (Bahgat & Khalifa, 2006), antioxidant (El Nezhawy *et al.*, 2009) and antiinflammatory (Khalifa *et al.*, 2008) activities. Tetralin derivative containing cyanoaceto-hydrazide was prepared as the title compound and its crystal structure is now reported.

The asymmetric unit of the title compound is shown in Fig. 1. The tetrahydrobenzene ring (C1–C6) adopts a half-boat conformation with puckering parameters (Cremer & Pople, 1975), $Q = 0.4695$ (16) Å, $\theta = 122.4$ (2)° and $\varphi = 308.6$ (2)°. The flap atom C3 deviates from the mean plane of C1/C2/C4–C6 by -0.6395 (16) Å. In the molecule, the mean plane of tetrahydronaphthalene ring system (C1–C10) forms a dihedral angle of 9.56 (6)° with the mean plane of cyano-acetohydrazide group [O1/N1–N3/C11–C13; maximum deviation = 0.045 (2) Å at atom C12].

In the crystal (Fig. 2), molecules are linked by a pair of N1—H1N1...O1 hydrogen bonds into an inversion dimer with an $R_2^2(8)$ ring motif. The dimers are further connected by C10—H10A...N3 hydrogen bonds into an infinite chain along [101]. The crystal packing also features C—H... π interaction (Table 1), involving Cg1 which is the centroid of C5–C10 ring.

Experimental

Equimolar amounts (0.01 mol) of tetralone and 2-cyanoacetohydrazide in dioxane (30 ml) were heated under reflux for 6 h. The mixture was then cooled at room temperature for overnight. The precipitated solid was filtered off, washed with ethanol, dried and crystallized from methanol to afford the title compound as colourless blocks with 73% abundance, *m.p.*: 183–185 °C.

Refinement

The atom H1N1 was located in a difference fourier map and refined freely [N1—H1N1 = 0.90 (2) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93 and 0.97 Å] and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication:

SHELXTL (Sheldrick, 2008) and *PLATON* (Spek, 2009).

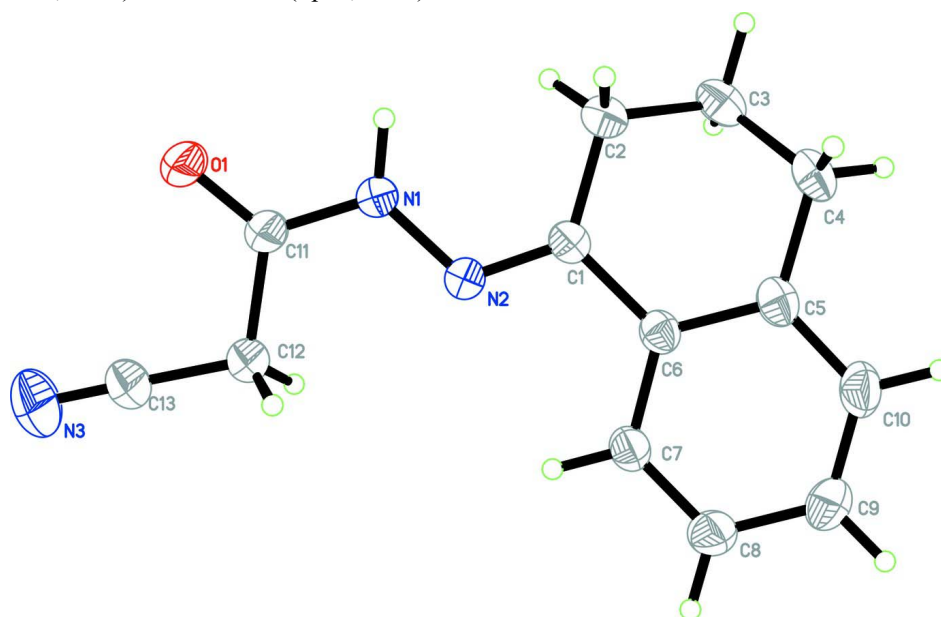


Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids.

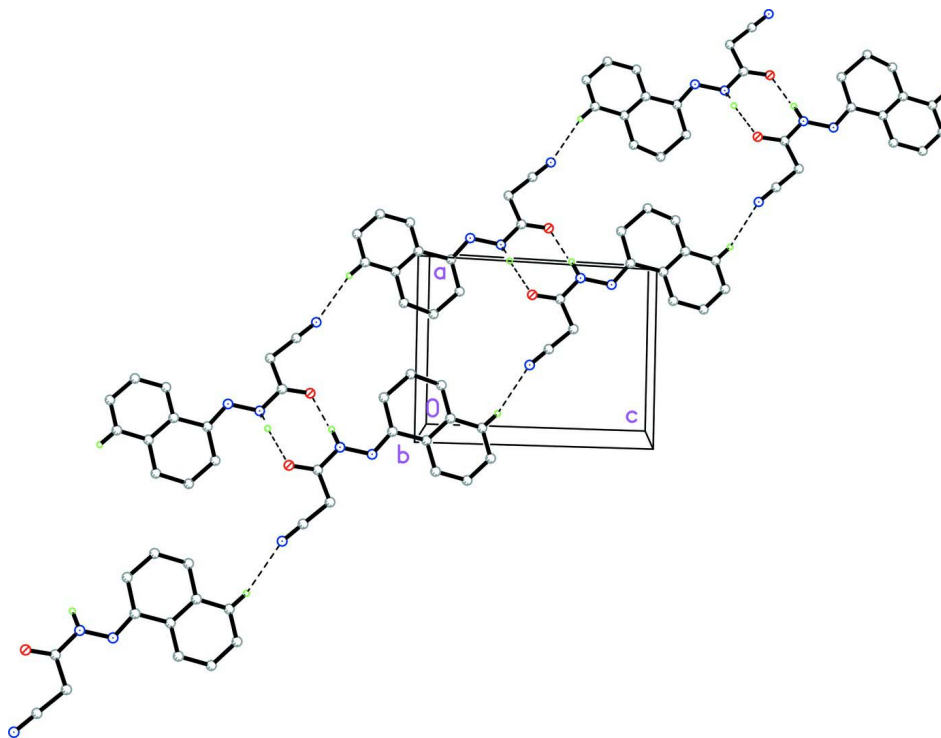


Figure 2

The crystal packing of the title compound. The dashed lines represent the hydrogen bonds. For clarity sake, hydrogen atoms not involved in hydrogen bonding have been omitted.

(E)-2-Cyano-N'-(1,2,3,4-tetrahydronaphthalen-1-ylidene)acetohydrazide

Crystal data

$C_{13}H_{13}N_3O$	$Z = 2$
$M_r = 227.26$	$F(000) = 240$
Triclinic, $P\bar{1}$	$D_x = 1.297 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$
$a = 7.6414 (1) \text{ \AA}$	Cell parameters from 2725 reflections
$b = 7.6748 (1) \text{ \AA}$	$\theta = 5.8\text{--}70.2^\circ$
$c = 10.5644 (2) \text{ \AA}$	$\mu = 0.69 \text{ mm}^{-1}$
$\alpha = 109.589 (1)^\circ$	$T = 296 \text{ K}$
$\beta = 91.405 (1)^\circ$	Block, colourless
$\gamma = 93.260 (1)^\circ$	$0.59 \times 0.51 \times 0.40 \text{ mm}$
$V = 582.13 (2) \text{ \AA}^3$	

Data collection

Bruker SMART APEXII CCD diffractometer	5749 measured reflections
Radiation source: fine-focus sealed tube	1898 independent reflections
Graphite monochromator	1762 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.017$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$\theta_{\text{max}} = 65.0^\circ$, $\theta_{\text{min}} = 5.8^\circ$
$T_{\text{min}} = 0.687$, $T_{\text{max}} = 0.771$	$h = -8 \rightarrow 8$
	$k = -7 \rightarrow 9$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.0904P]$
$wR(F^2) = 0.120$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1898 reflections	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
159 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
0 restraints	Extinction correction: SHELXTL (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.030 (3)
Secondary atom site location: difference Fourier map	

Special details

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.

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.19129 (16)	0.4167 (2)	0.53245 (10)	0.0798 (4)
N1	0.07938 (15)	0.36666 (18)	0.32370 (11)	0.0530 (3)

N2	0.10531 (14)	0.29801 (15)	0.18782 (10)	0.0456 (3)
N3	0.5764 (3)	0.2338 (4)	0.5360 (2)	0.1264 (9)
C1	-0.02329 (16)	0.29510 (17)	0.10641 (13)	0.0419 (3)
C2	-0.20191 (18)	0.3606 (2)	0.14809 (15)	0.0531 (4)
H2A	-0.1954	0.4950	0.1788	0.064*
H2B	-0.2357	0.3218	0.2230	0.064*
C3	-0.34209 (18)	0.2862 (2)	0.03515 (16)	0.0590 (4)
H3A	-0.3665	0.1544	0.0168	0.071*
H3B	-0.4494	0.3470	0.0632	0.071*
C4	-0.28501 (19)	0.3183 (2)	-0.09133 (16)	0.0583 (4)
H4A	-0.2742	0.4504	-0.0759	0.070*
H4B	-0.3738	0.2621	-0.1629	0.070*
C5	-0.11171 (18)	0.23730 (19)	-0.13436 (14)	0.0491 (4)
C6	0.01244 (16)	0.22607 (16)	-0.03809 (12)	0.0416 (3)
C7	0.17256 (17)	0.15242 (19)	-0.08101 (14)	0.0478 (3)
H7A	0.2555	0.1441	-0.0177	0.057*
C8	0.2098 (2)	0.0923 (2)	-0.21424 (15)	0.0575 (4)
H8A	0.3170	0.0438	-0.2407	0.069*
C9	0.0876 (2)	0.1038 (2)	-0.30948 (15)	0.0636 (4)
H9A	0.1122	0.0629	-0.4001	0.076*
C10	-0.0706 (2)	0.1761 (2)	-0.26907 (15)	0.0613 (4)
H10A	-0.1520	0.1842	-0.3334	0.074*
C11	0.20438 (19)	0.3556 (2)	0.41121 (14)	0.0547 (4)
C12	0.3655 (2)	0.2592 (2)	0.35112 (15)	0.0619 (4)
H12A	0.3304	0.1361	0.2892	0.074*
H12B	0.4251	0.3284	0.3011	0.074*
C13	0.4837 (2)	0.2454 (3)	0.45556 (17)	0.0712 (5)
H1N1	-0.010 (3)	0.434 (3)	0.3642 (19)	0.078 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0795 (8)	0.1259 (11)	0.0388 (6)	0.0515 (7)	0.0104 (5)	0.0263 (6)
N1	0.0473 (7)	0.0739 (8)	0.0390 (6)	0.0203 (6)	0.0055 (5)	0.0178 (5)
N2	0.0432 (6)	0.0558 (7)	0.0378 (6)	0.0092 (5)	0.0022 (4)	0.0148 (5)
N3	0.0968 (14)	0.189 (2)	0.0923 (14)	0.0546 (15)	-0.0274 (11)	0.0414 (14)
C1	0.0377 (7)	0.0438 (7)	0.0456 (7)	0.0036 (5)	0.0000 (5)	0.0167 (5)
C2	0.0420 (7)	0.0643 (9)	0.0538 (8)	0.0104 (6)	0.0054 (6)	0.0195 (7)
C3	0.0374 (7)	0.0665 (9)	0.0744 (10)	0.0053 (6)	-0.0015 (6)	0.0255 (8)
C4	0.0441 (8)	0.0667 (9)	0.0653 (9)	0.0060 (6)	-0.0122 (6)	0.0245 (7)
C5	0.0452 (7)	0.0495 (7)	0.0512 (8)	0.0002 (6)	-0.0089 (6)	0.0164 (6)
C6	0.0395 (7)	0.0401 (6)	0.0429 (7)	0.0007 (5)	-0.0037 (5)	0.0117 (5)
C7	0.0438 (7)	0.0517 (7)	0.0450 (7)	0.0076 (6)	-0.0041 (5)	0.0122 (6)
C8	0.0544 (8)	0.0603 (9)	0.0503 (8)	0.0106 (7)	0.0047 (6)	0.0078 (7)
C9	0.0711 (10)	0.0723 (10)	0.0396 (7)	0.0049 (8)	-0.0002 (7)	0.0089 (7)
C10	0.0632 (9)	0.0711 (10)	0.0466 (8)	0.0019 (7)	-0.0144 (7)	0.0174 (7)
C11	0.0542 (8)	0.0729 (9)	0.0398 (7)	0.0209 (7)	0.0061 (6)	0.0197 (6)
C12	0.0528 (8)	0.0840 (11)	0.0442 (8)	0.0228 (7)	0.0006 (6)	0.0127 (7)
C13	0.0574 (9)	0.0923 (12)	0.0596 (9)	0.0241 (8)	-0.0055 (8)	0.0175 (9)

Geometric parameters (Å, °)

O1—C11	1.2163 (17)	C4—H4B	0.9700
N1—C11	1.3389 (18)	C5—C10	1.391 (2)
N1—N2	1.3770 (15)	C5—C6	1.4009 (18)
N1—H1N1	0.90 (2)	C6—C7	1.3986 (19)
N2—C1	1.2841 (16)	C7—C8	1.369 (2)
N3—C13	1.122 (2)	C7—H7A	0.9300
C1—C6	1.4766 (18)	C8—C9	1.383 (2)
C1—C2	1.5061 (18)	C8—H8A	0.9300
C2—C3	1.519 (2)	C9—C10	1.376 (2)
C2—H2A	0.9700	C9—H9A	0.9300
C2—H2B	0.9700	C10—H10A	0.9300
C3—C4	1.508 (2)	C11—C12	1.5140 (19)
C3—H3A	0.9700	C12—C13	1.444 (2)
C3—H3B	0.9700	C12—H12A	0.9700
C4—C5	1.510 (2)	C12—H12B	0.9700
C4—H4A	0.9700		
C11—N1—N2	119.64 (11)	C6—C5—C4	120.20 (13)
C11—N1—H1N1	112.9 (12)	C7—C6—C5	118.82 (12)
N2—N1—H1N1	127.0 (12)	C7—C6—C1	120.57 (11)
C1—N2—N1	117.97 (11)	C5—C6—C1	120.59 (11)
N2—C1—C6	116.02 (11)	C8—C7—C6	121.36 (12)
N2—C1—C2	124.94 (12)	C8—C7—H7A	119.3
C6—C1—C2	119.01 (11)	C6—C7—H7A	119.3
C1—C2—C3	112.97 (12)	C7—C8—C9	119.94 (13)
C1—C2—H2A	109.0	C7—C8—H8A	120.0
C3—C2—H2A	109.0	C9—C8—H8A	120.0
C1—C2—H2B	109.0	C10—C9—C8	119.50 (14)
C3—C2—H2B	109.0	C10—C9—H9A	120.3
H2A—C2—H2B	107.8	C8—C9—H9A	120.3
C4—C3—C2	111.41 (12)	C9—C10—C5	121.62 (13)
C4—C3—H3A	109.3	C9—C10—H10A	119.2
C2—C3—H3A	109.3	C5—C10—H10A	119.2
C4—C3—H3B	109.3	O1—C11—N1	122.97 (13)
C2—C3—H3B	109.3	O1—C11—C12	120.79 (13)
H3A—C3—H3B	108.0	N1—C11—C12	116.23 (12)
C3—C4—C5	111.56 (12)	C13—C12—C11	110.54 (13)
C3—C4—H4A	109.3	C13—C12—H12A	109.5
C5—C4—H4A	109.3	C11—C12—H12A	109.5
C3—C4—H4B	109.3	C13—C12—H12B	109.5
C5—C4—H4B	109.3	C11—C12—H12B	109.5
H4A—C4—H4B	108.0	H12A—C12—H12B	108.1
C10—C5—C6	118.76 (13)	N3—C13—C12	179.4 (2)
C10—C5—C4	121.04 (12)		
C11—N1—N2—C1	173.62 (13)	C2—C1—C6—C7	-175.60 (12)
N1—N2—C1—C6	178.06 (11)	N2—C1—C6—C5	-172.51 (11)
N1—N2—C1—C2	-0.3 (2)	C2—C1—C6—C5	5.95 (18)

N2—C1—C2—C3	-161.63 (13)	C5—C6—C7—C8	0.2 (2)
C6—C1—C2—C3	20.06 (18)	C1—C6—C7—C8	-178.26 (12)
C1—C2—C3—C4	-50.51 (17)	C6—C7—C8—C9	0.0 (2)
C2—C3—C4—C5	55.23 (17)	C7—C8—C9—C10	0.1 (2)
C3—C4—C5—C10	151.26 (14)	C8—C9—C10—C5	-0.4 (3)
C3—C4—C5—C6	-30.03 (18)	C6—C5—C10—C9	0.6 (2)
C10—C5—C6—C7	-0.5 (2)	C4—C5—C10—C9	179.32 (15)
C4—C5—C6—C7	-179.24 (12)	N2—N1—C11—O1	178.53 (15)
C10—C5—C6—C1	177.98 (12)	N2—N1—C11—C12	-2.2 (2)
C4—C5—C6—C1	-0.76 (19)	O1—C11—C12—C13	3.3 (2)
N2—C1—C6—C7	5.94 (18)	N1—C11—C12—C13	-175.97 (15)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C5–C10 ring.

<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>
N1—H1N1...O1 ⁱ	0.91 (2)	1.96 (2)	2.8640 (17)	174.7 (19)
C10—H10A...N3 ⁱⁱ	0.93	2.58	3.491 (3)	167
C2—H2A...Cg1 ⁱⁱⁱ	0.97	2.80	3.6775 (17)	152

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