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N'-[(1*E*,2*E*)-3,7-Dimethylocta-2,6-dien-1-ylidene]pyridine-4-carbohydrazide

Mashooq A. Bhat,^a Hatem A. Abdel-Aziz,^a Hazem A. Ghabbour,^a Madhukar Hemamalini^b and Hoong-Kun Fun^{b*‡}

^aDepartment of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, PO Box 2457, Riyadh 11451, Saudi Arabia, and ^bX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

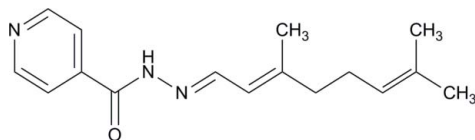
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.049; wR factor = 0.154; data-to-parameter ratio = 12.5.

In the title compound, $\text{C}_{16}\text{H}_{21}\text{N}_3\text{O}$, the molecule adopts an *E* conformation about the central $\text{C}=\text{N}$ double bond. The 2-methylpent-2-ene group is disordered over two sets of sites, with a refined occupancy ratio of 0.785 (8):0.215 (8). The dihedral angle between the essentially planar [the r.m.s. value for the major component is 0.021 (7) and its maximum deviation is 0.025 (4) Å; the r.m.s. value for the minor component is 0.03 (4) and its maximum deviation is 0.05 (3) Å] major and minor components of the 2-methylbut-2-ene group is 35.9 (13)°. In the crystal, $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules, with the same O atom acting as the acceptor. This results in $C_1^1(4)$ and $C_1^1(5)$ [001] chains.

Related literature

For details and the biological activity of isoniazide, see: Janin (2007); Maccari *et al.* (2005); Slayden & Barry (2000); Hearn *et al.* (2009); Tripathi *et al.* (2011). For related structures, see: Naveenkumar *et al.* (2010); Jiang *et al.* (2009); Khan *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



‡ College of Pharmacy (Visiting Professor), King Saud University, PO Box 2457, Riyadh 11451, Saudi Arabia. Thomson Reuters ResearcherID: A-3561-2009.

Experimental

Crystal data

$\text{C}_{16}\text{H}_{21}\text{N}_3\text{O}$
 $M_r = 271.36$
 Monoclinic, $P2_1/c$
 $a = 17.5415$ (8) Å
 $b = 12.0708$ (6) Å
 $c = 7.8430$ (4) Å
 $\beta = 101.854$ (3)°
 $V = 1625.26$ (14) Å³
 $Z = 4$
 Cu $K\alpha$ radiation
 $\mu = 0.56$ mm⁻¹
 $T = 296$ K
 $0.90 \times 0.27 \times 0.17$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.633$, $T_{\max} = 0.912$
 16548 measured reflections
 2978 independent reflections
 2376 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.154$
 $S = 1.03$
 2978 reflections
 239 parameters
 12 restraints
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N3}-\text{H1N3}\cdots\text{O1}^i$ | 0.873 (17) | 2.052 (17) | 2.9167 (18) | 170.8 (16) |
| $\text{C4}-\text{H4A}\cdots\text{O1}^i$ | 0.93 | 2.53 | 3.251 (2) | 135 |

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

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: LH5421).

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supporting information

Acta Cryst. (2012). E68, o1144–o1145 [https://doi.org/10.1107/S1600536812009075]

***N'*-[(1*E*,2*E*)-3,7-Dimethylocta-2,6-dien-1-ylidene]pyridine-4-carbohydrazide**

Mashooq A. Bhat, Hatem A. Abdel-Aziz, Hazem A. Ghabbour, Madhukar Hemamalini and Hoong-Kun Fun

S1. Comment

In the search of new compounds, isoniazid derivatives have been found to possess potential tuberculostatic activity (Janin, 2007; Maccari *et al.*, 2005; Slayden & Barry, 2000; Hearn *et al.*, 2009; Tripathi *et al.*, 2011). The crystal structures of (*E*)-*N'*-(2-Benzoyloxybenzylidene)isonicotinohydrazide methanol solvate monohydrate (Naveenkumar *et al.*, 2010), *N'*-(1-Phenylethylidene)isonicotinohydrazide (Jiang *et al.*, 2009) and *N'*-(4-Bromophenylsulfonyl)isonicotinohydrazide (Khan *et al.*, 2009) have been reported in the literature. Here, we present the crystal structure of the title compound, (I).

The asymmetric unit of the title compound is shown in Fig. 1. The molecule adopts an *E* configuration about the central C7=N2 double bond. The 2-methylpent-2-ene group is disordered over two sets of sites, with a refined occupancy ratio of 0.785 (8):0.215 (8). The dihedral angles between the major and minor components of the 2-methylbut-2-ene (C11–C15:C11A–C15A) group is 35.9 (13)°.

In the crystal, Fig. 2, the adjacent molecules are connected *via* bifurcated N—H···O and C—H···O hydrogen bonds (Table 1), generating $R^1_2(7)$ ring motifs (Bernstein *et al.*, 1995), resulting in supramolecular [001] chains.

S2. Experimental

The title compound was prepared by the reaction of citral, 3,7-dimethylocta-2,6-dienal (0.15 g, 1 mmol) with isoniazid (0.14 g, 1 mmol) in ETOH/H₂O (3:1, v/v, 10 mL). After stirring for 3 h at room temperature, the resulting mixture was concentrated under reduced pressure. The residue washed with cold ethyl alcohol and then with ethyl ether to afford the title compound. Colorless blocks of the latter compound suitable for X-ray structure determination were recrystallized from ETOH by the slow evaporation of the solvent at room temperature.

S3. Refinement

Atom H1N3 was located from a difference Fourier maps and refined freely [N—H = 0.873 (18) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93–0.97 Å] and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl groups. The 2-methylpent-2-ene group is disordered over two sets of sites, with a refined occupancy ratio of 0.785 (8):0.215 (8).

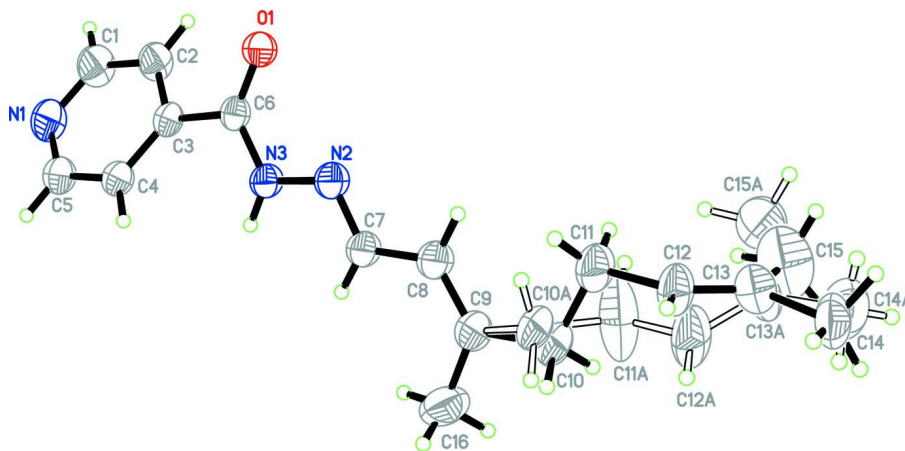


Figure 1

The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids. Open bonds represent disordered components.

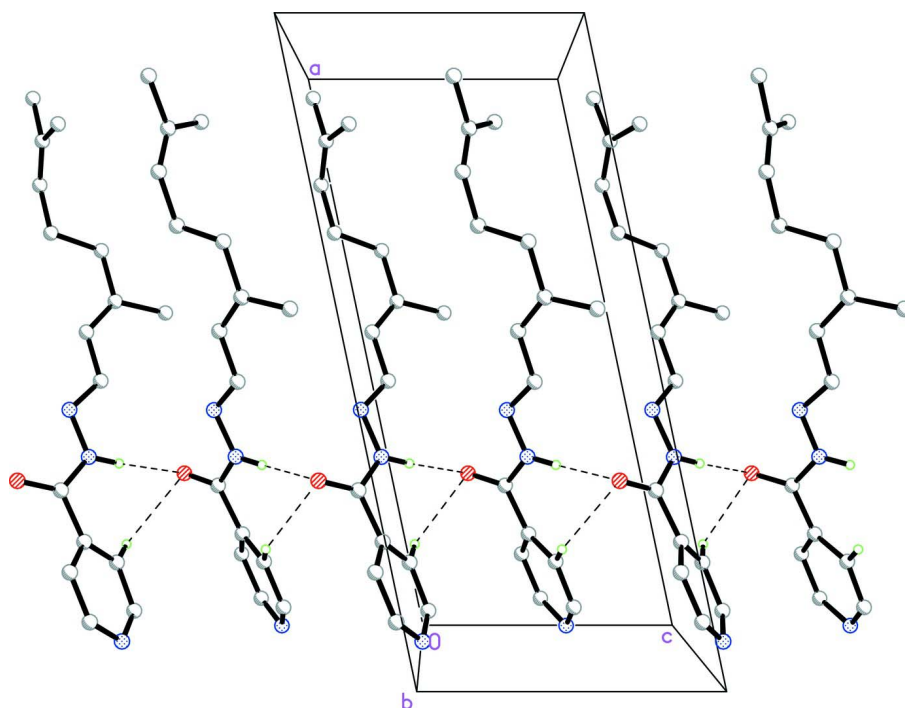


Figure 2

The crystal packing of the title compound (I). Hydrogen bonds are shown as dashed lines. The disorder is not shown.

N'-[(1*E*,2*E*)-3,7-Dimethylocta-2,6-dien-1-ylidene]pyridine- 4-carbohydrazide

Crystal data

$C_{16}H_{21}N_3O$

$M_r = 271.36$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 17.5415\ (8)\ \text{\AA}$

$b = 12.0708\ (6)\ \text{\AA}$

$c = 7.8430\ (4)\ \text{\AA}$

$\beta = 101.854\ (3)^\circ$

$V = 1625.26\ (14)\ \text{\AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.109\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$
 Cell parameters from 1053 reflections
 $\theta = 11.3\text{--}69.5^\circ$
 $\mu = 0.56 \text{ mm}^{-1}$

$T = 296 \text{ K}$
 Block, colourless
 $0.90 \times 0.27 \times 0.17 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.633$, $T_{\max} = 0.912$

16548 measured reflections
 2978 independent reflections
 2376 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\max} = 69.7^\circ$, $\theta_{\min} = 5.2^\circ$
 $h = -16 \rightarrow 21$
 $k = -14 \rightarrow 14$
 $l = -9 \rightarrow 7$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.154$
 $S = 1.03$
 2978 reflections
 239 parameters
 12 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
 $w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 0.2272P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.14 \text{ e \AA}^{-3}$
 Extinction correction: SHELXTL (Sheldrick,
 2008), $F_c^* = kFc[1 + 0.001xFe^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0032 (6)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------------|--------------|----------------------------------|-----------|
| O1 | 0.30512 (6) | 0.61845 (10) | 0.31148 (14) | 0.0640 (3) | |
| N1 | 0.06078 (9) | 0.62725 (17) | 0.5444 (3) | 0.0897 (5) | |
| N2 | 0.40983 (7) | 0.76093 (12) | 0.49025 (19) | 0.0656 (4) | |
| N3 | 0.33659 (7) | 0.74425 (12) | 0.52777 (18) | 0.0602 (4) | |
| C1 | 0.09771 (12) | 0.54801 (19) | 0.4767 (3) | 0.0905 (7) | |
| H1A | 0.0724 | 0.4805 | 0.4511 | 0.109* | |
| C2 | 0.17110 (10) | 0.55920 (16) | 0.4418 (3) | 0.0746 (5) | |
| H2A | 0.1942 | 0.5007 | 0.3941 | 0.090* | |
| C3 | 0.20985 (8) | 0.65855 (13) | 0.47852 (19) | 0.0558 (4) | |
| C4 | 0.17217 (9) | 0.74139 (15) | 0.5489 (2) | 0.0686 (5) | |
| H4A | 0.1959 | 0.8099 | 0.5756 | 0.082* | |

| | | | | | |
|------|--------------|--------------|--------------|-------------|-----------|
| C5 | 0.09864 (10) | 0.72159 (19) | 0.5792 (3) | 0.0822 (6) | |
| H5A | 0.0742 | 0.7785 | 0.6274 | 0.099* | |
| C6 | 0.28809 (8) | 0.67137 (13) | 0.43211 (19) | 0.0534 (4) | |
| C7 | 0.45447 (9) | 0.82229 (15) | 0.6011 (2) | 0.0645 (4) | |
| H7A | 0.4366 | 0.8509 | 0.6957 | 0.077* | |
| C8 | 0.53221 (10) | 0.84735 (17) | 0.5801 (3) | 0.0746 (5) | |
| H8A | 0.5485 | 0.8169 | 0.4846 | 0.089* | |
| C9 | 0.58251 (11) | 0.91090 (18) | 0.6876 (3) | 0.0837 (6) | |
| C10 | 0.66541 (16) | 0.9344 (4) | 0.6696 (6) | 0.0871 (10) | 0.785 (8) |
| H10A | 0.6713 | 1.0139 | 0.6600 | 0.105* | 0.785 (8) |
| H10B | 0.7002 | 0.9109 | 0.7762 | 0.105* | 0.785 (8) |
| C11 | 0.69190 (18) | 0.8806 (3) | 0.5187 (5) | 0.0900 (11) | 0.785 (8) |
| H11A | 0.6929 | 0.8008 | 0.5341 | 0.108* | 0.785 (8) |
| H11B | 0.6550 | 0.8975 | 0.4118 | 0.108* | 0.785 (8) |
| C12 | 0.77113 (18) | 0.9196 (3) | 0.5033 (6) | 0.0828 (10) | 0.785 (8) |
| H12A | 0.7710 | 0.9863 | 0.4439 | 0.099* | 0.785 (8) |
| C13 | 0.8396 (4) | 0.8782 (5) | 0.5571 (15) | 0.0811 (19) | 0.785 (8) |
| C14 | 0.9134 (3) | 0.9352 (6) | 0.5393 (11) | 0.1003 (15) | 0.785 (8) |
| H14A | 0.9011 | 1.0045 | 0.4798 | 0.150* | 0.785 (8) |
| H14B | 0.9416 | 0.8891 | 0.4739 | 0.150* | 0.785 (8) |
| H14C | 0.9448 | 0.9486 | 0.6529 | 0.150* | 0.785 (8) |
| C15 | 0.8582 (5) | 0.7702 (6) | 0.6537 (11) | 0.162 (3) | 0.785 (8) |
| H15A | 0.8121 | 0.7420 | 0.6863 | 0.242* | 0.785 (8) |
| H15B | 0.8977 | 0.7823 | 0.7564 | 0.242* | 0.785 (8) |
| H15C | 0.8766 | 0.7175 | 0.5797 | 0.242* | 0.785 (8) |
| C10A | 0.6475 (6) | 0.9475 (14) | 0.5992 (19) | 0.0871 (10) | 0.215 (8) |
| H10C | 0.6314 | 0.9371 | 0.4743 | 0.105* | 0.215 (8) |
| H10D | 0.6577 | 1.0258 | 0.6210 | 0.105* | 0.215 (8) |
| C11A | 0.7184 (8) | 0.8848 (17) | 0.663 (4) | 0.169 (13) | 0.215 (8) |
| H11C | 0.7112 | 0.8104 | 0.6156 | 0.203* | 0.215 (8) |
| H11D | 0.7257 | 0.8787 | 0.7887 | 0.203* | 0.215 (8) |
| C12A | 0.7911 (7) | 0.9321 (12) | 0.620 (4) | 0.123 (7) | 0.215 (8) |
| H12B | 0.7971 | 1.0081 | 0.6089 | 0.148* | 0.215 (8) |
| C13A | 0.8464 (13) | 0.8625 (17) | 0.599 (5) | 0.080 (8) | 0.215 (8) |
| C14A | 0.9212 (14) | 0.901 (3) | 0.562 (6) | 0.170 (16) | 0.215 (8) |
| H14D | 0.9258 | 0.9793 | 0.5797 | 0.255* | 0.215 (8) |
| H14E | 0.9232 | 0.8837 | 0.4434 | 0.255* | 0.215 (8) |
| H14F | 0.9633 | 0.8641 | 0.6388 | 0.255* | 0.215 (8) |
| C15A | 0.8294 (13) | 0.7391 (13) | 0.588 (4) | 0.137 (8) | 0.215 (8) |
| H15D | 0.7743 | 0.7273 | 0.5742 | 0.206* | 0.215 (8) |
| H15E | 0.8562 | 0.7036 | 0.6925 | 0.206* | 0.215 (8) |
| H15F | 0.8468 | 0.7084 | 0.4895 | 0.206* | 0.215 (8) |
| C16 | 0.56438 (16) | 0.9641 (3) | 0.8453 (4) | 0.1287 (12) | |
| H16A | 0.5169 | 0.9337 | 0.8683 | 0.193* | |
| H16B | 0.5584 | 1.0424 | 0.8265 | 0.193* | |
| H16C | 0.6061 | 0.9505 | 0.9432 | 0.193* | |
| H1N3 | 0.3250 (10) | 0.7786 (15) | 0.617 (2) | 0.063 (5)* | |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0566 (6) | 0.0690 (7) | 0.0683 (7) | -0.0046 (5) | 0.0170 (5) | -0.0065 (5) |
| N1 | 0.0508 (8) | 0.1053 (13) | 0.1165 (14) | -0.0154 (8) | 0.0259 (8) | -0.0068 (10) |
| N2 | 0.0505 (7) | 0.0773 (9) | 0.0744 (8) | -0.0136 (6) | 0.0255 (6) | -0.0094 (7) |
| N3 | 0.0465 (7) | 0.0721 (9) | 0.0658 (8) | -0.0116 (6) | 0.0201 (6) | -0.0093 (6) |
| C1 | 0.0625 (10) | 0.0804 (13) | 0.1331 (18) | -0.0218 (10) | 0.0307 (11) | -0.0046 (12) |
| C2 | 0.0580 (9) | 0.0658 (10) | 0.1026 (13) | -0.0102 (8) | 0.0223 (9) | -0.0010 (9) |
| C3 | 0.0449 (7) | 0.0622 (9) | 0.0593 (8) | -0.0041 (6) | 0.0088 (6) | 0.0055 (7) |
| C4 | 0.0483 (8) | 0.0737 (11) | 0.0836 (11) | -0.0076 (7) | 0.0132 (7) | -0.0115 (8) |
| C5 | 0.0489 (9) | 0.0978 (14) | 0.1012 (14) | -0.0048 (9) | 0.0183 (9) | -0.0194 (11) |
| C6 | 0.0462 (7) | 0.0559 (8) | 0.0584 (8) | -0.0027 (6) | 0.0113 (6) | 0.0051 (6) |
| C7 | 0.0512 (8) | 0.0720 (10) | 0.0741 (10) | -0.0121 (7) | 0.0217 (7) | -0.0097 (8) |
| C8 | 0.0554 (9) | 0.0845 (12) | 0.0895 (12) | -0.0165 (8) | 0.0281 (8) | -0.0174 (9) |
| C9 | 0.0561 (10) | 0.0823 (13) | 0.1158 (15) | -0.0172 (9) | 0.0251 (10) | -0.0202 (11) |
| C10 | 0.0477 (14) | 0.0936 (18) | 0.117 (3) | -0.0193 (15) | 0.0097 (16) | -0.015 (2) |
| C11 | 0.0575 (16) | 0.111 (2) | 0.107 (2) | -0.0183 (15) | 0.0294 (15) | -0.0033 (18) |
| C12 | 0.0540 (15) | 0.0843 (19) | 0.113 (2) | -0.0069 (13) | 0.0249 (15) | 0.0191 (18) |
| C13 | 0.078 (3) | 0.077 (3) | 0.093 (4) | 0.007 (2) | 0.026 (2) | 0.018 (3) |
| C14 | 0.0549 (18) | 0.110 (4) | 0.140 (3) | 0.004 (2) | 0.030 (2) | 0.001 (3) |
| C15 | 0.162 (6) | 0.127 (5) | 0.209 (7) | 0.022 (4) | 0.068 (5) | 0.075 (5) |
| C10A | 0.0477 (14) | 0.0936 (18) | 0.117 (3) | -0.0193 (15) | 0.0097 (16) | -0.015 (2) |
| C11A | 0.081 (9) | 0.146 (15) | 0.29 (3) | 0.031 (9) | 0.064 (14) | 0.12 (2) |
| C12A | 0.064 (7) | 0.086 (8) | 0.22 (2) | -0.013 (6) | 0.039 (11) | 0.026 (12) |
| C13A | 0.072 (8) | 0.069 (9) | 0.11 (2) | 0.023 (6) | 0.037 (10) | 0.034 (8) |
| C14A | 0.095 (14) | 0.12 (2) | 0.28 (4) | 0.012 (12) | 0.001 (16) | -0.03 (2) |
| C15A | 0.121 (14) | 0.070 (9) | 0.20 (2) | 0.029 (9) | -0.008 (12) | -0.009 (10) |
| C16 | 0.0951 (17) | 0.160 (3) | 0.137 (2) | -0.0471 (18) | 0.0378 (15) | -0.071 (2) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|---------|-------------|-----------|------------|
| O1—C6 | 1.2283 (18) | C12—C13 | 1.290 (7) |
| N1—C5 | 1.318 (3) | C12—H12A | 0.9300 |
| N1—C1 | 1.326 (3) | C13—C14 | 1.497 (6) |
| N2—C7 | 1.280 (2) | C13—C15 | 1.510 (7) |
| N2—N3 | 1.3903 (17) | C14—H14A | 0.9600 |
| N3—C6 | 1.341 (2) | C14—H14B | 0.9600 |
| N3—H1N3 | 0.873 (18) | C14—H14C | 0.9600 |
| C1—C2 | 1.377 (2) | C15—H15A | 0.9600 |
| C1—H1A | 0.9300 | C15—H15B | 0.9600 |
| C2—C3 | 1.379 (2) | C15—H15C | 0.9600 |
| C2—H2A | 0.9300 | C10A—C11A | 1.454 (14) |
| C3—C4 | 1.375 (2) | C10A—H10C | 0.9700 |
| C3—C6 | 1.4985 (18) | C10A—H10D | 0.9700 |
| C4—C5 | 1.380 (2) | C11A—C12A | 1.497 (13) |
| C4—H4A | 0.9300 | C11A—H11C | 0.9700 |
| C5—H5A | 0.9300 | C11A—H11D | 0.9700 |

| | | | |
|-------------|-------------|----------------|------------|
| C7—C8 | 1.439 (2) | C12A—C13A | 1.320 (16) |
| C7—H7A | 0.9300 | C12A—H12B | 0.9300 |
| C8—C9 | 1.331 (3) | C13A—C14A | 1.474 (16) |
| C8—H8A | 0.9300 | C13A—C15A | 1.518 (17) |
| C9—C16 | 1.485 (3) | C14A—H14D | 0.9600 |
| C9—C10 | 1.516 (2) | C14A—H14E | 0.9600 |
| C9—C10A | 1.517 (3) | C14A—H14F | 0.9600 |
| C10—C11 | 1.505 (4) | C15A—H15D | 0.9600 |
| C10—H10A | 0.9700 | C15A—H15E | 0.9600 |
| C10—H10B | 0.9700 | C15A—H15F | 0.9600 |
| C11—C12 | 1.496 (4) | C16—H16A | 0.9600 |
| C11—H11A | 0.9700 | C16—H16B | 0.9600 |
| C11—H11B | 0.9700 | C16—H16C | 0.9600 |
| | | | |
| C5—N1—C1 | 116.07 (15) | C12—C13—C15 | 126.1 (5) |
| C7—N2—N3 | 113.78 (13) | C14—C13—C15 | 110.0 (6) |
| C6—N3—N2 | 118.96 (13) | C13—C14—H14A | 109.5 |
| C6—N3—H1N3 | 122.3 (12) | C13—C14—H14B | 109.5 |
| N2—N3—H1N3 | 118.6 (12) | H14A—C14—H14B | 109.5 |
| N1—C1—C2 | 124.24 (18) | C13—C14—H14C | 109.5 |
| N1—C1—H1A | 117.9 | H14A—C14—H14C | 109.5 |
| C2—C1—H1A | 117.9 | H14B—C14—H14C | 109.5 |
| C1—C2—C3 | 118.98 (18) | C13—C15—H15A | 109.5 |
| C1—C2—H2A | 120.5 | C13—C15—H15B | 109.5 |
| C3—C2—H2A | 120.5 | H15A—C15—H15B | 109.5 |
| C4—C3—C2 | 117.33 (14) | C13—C15—H15C | 109.5 |
| C4—C3—C6 | 124.20 (14) | H15A—C15—H15C | 109.5 |
| C2—C3—C6 | 118.40 (15) | H15B—C15—H15C | 109.5 |
| C3—C4—C5 | 119.13 (17) | C11A—C10A—C9 | 111.1 (10) |
| C3—C4—H4A | 120.4 | C11A—C10A—H10C | 109.4 |
| C5—C4—H4A | 120.4 | C9—C10A—H10C | 109.4 |
| N1—C5—C4 | 124.25 (18) | C11A—C10A—H10D | 109.4 |
| N1—C5—H5A | 117.9 | C9—C10A—H10D | 109.4 |
| C4—C5—H5A | 117.9 | H10C—C10A—H10D | 108.0 |
| O1—C6—N3 | 123.05 (13) | C10A—C11A—C12A | 115.6 (10) |
| O1—C6—C3 | 120.88 (13) | C10A—C11A—H11C | 108.4 |
| N3—C6—C3 | 116.06 (13) | C12A—C11A—H11C | 108.4 |
| N2—C7—C8 | 120.42 (15) | C10A—C11A—H11D | 108.4 |
| N2—C7—H7A | 119.8 | C12A—C11A—H11D | 108.4 |
| C8—C7—H7A | 119.8 | H11C—C11A—H11D | 107.5 |
| C9—C8—C7 | 124.73 (17) | C13A—C12A—C11A | 117.9 (14) |
| C9—C8—H8A | 117.6 | C13A—C12A—H12B | 121.0 |
| C7—C8—H8A | 117.6 | C11A—C12A—H12B | 121.0 |
| C8—C9—C16 | 123.28 (17) | C12A—C13A—C14A | 122.2 (17) |
| C8—C9—C10 | 124.9 (2) | C12A—C13A—C15A | 119.4 (16) |
| C16—C9—C10 | 111.7 (2) | C14A—C13A—C15A | 118.0 (18) |
| C8—C9—C10A | 110.0 (7) | C13A—C14A—H14D | 109.5 |
| C16—C9—C10A | 124.4 (7) | C13A—C14A—H14E | 109.5 |

| | | | |
|---------------|--------------|---------------------|-------------|
| C10—C9—C10A | 22.7 (5) | H14D—C14A—H14E | 109.5 |
| C11—C10—C9 | 116.7 (3) | C13A—C14A—H14F | 109.5 |
| C11—C10—H10A | 108.1 | H14D—C14A—H14F | 109.5 |
| C9—C10—H10A | 108.1 | H14E—C14A—H14F | 109.5 |
| C11—C10—H10B | 108.1 | C13A—C15A—H15D | 109.5 |
| C9—C10—H10B | 108.1 | C13A—C15A—H15E | 109.5 |
| H10A—C10—H10B | 107.3 | H15D—C15A—H15E | 109.5 |
| C12—C11—C10 | 111.7 (3) | C13A—C15A—H15F | 109.5 |
| C12—C11—H11A | 109.3 | H15D—C15A—H15F | 109.5 |
| C10—C11—H11A | 109.3 | H15E—C15A—H15F | 109.5 |
| C12—C11—H11B | 109.3 | C9—C16—H16A | 109.5 |
| C10—C11—H11B | 109.3 | C9—C16—H16B | 109.5 |
| H11A—C11—H11B | 107.9 | H16A—C16—H16B | 109.5 |
| C13—C12—C11 | 132.1 (4) | C9—C16—H16C | 109.5 |
| C13—C12—H12A | 113.9 | H16A—C16—H16C | 109.5 |
| C11—C12—H12A | 113.9 | H16B—C16—H16C | 109.5 |
| C12—C13—C14 | 123.8 (6) | | |
| C7—N2—N3—C6 | -172.49 (15) | C7—C8—C9—C16 | 0.4 (4) |
| C5—N1—C1—C2 | -0.2 (4) | C7—C8—C9—C10 | 177.8 (3) |
| N1—C1—C2—C3 | 0.0 (4) | C7—C8—C9—C10A | -162.8 (6) |
| C1—C2—C3—C4 | 0.0 (3) | C8—C9—C10—C11 | -0.3 (6) |
| C1—C2—C3—C6 | -177.16 (18) | C16—C9—C10—C11 | 177.4 (4) |
| C2—C3—C4—C5 | 0.1 (3) | C10A—C9—C10—C11 | -54.2 (19) |
| C6—C3—C4—C5 | 177.14 (16) | C9—C10—C11—C12 | 173.7 (3) |
| C1—N1—C5—C4 | 0.3 (3) | C10—C11—C12—C13 | 96.6 (9) |
| C3—C4—C5—N1 | -0.3 (3) | C11—C12—C13—C14 | -175.3 (6) |
| N2—N3—C6—O1 | -0.3 (2) | C11—C12—C13—C15 | 1.4 (15) |
| N2—N3—C6—C3 | -179.34 (13) | C8—C9—C10A—C11A | -103 (2) |
| C4—C3—C6—O1 | -149.52 (17) | C16—C9—C10A—C11A | 94 (2) |
| C2—C3—C6—O1 | 27.5 (2) | C10—C9—C10A—C11A | 31.7 (18) |
| C4—C3—C6—N3 | 29.5 (2) | C9—C10A—C11A—C12A | -165.1 (17) |
| C2—C3—C6—N3 | -153.47 (16) | C10A—C11A—C12A—C13A | -149 (3) |
| N3—N2—C7—C8 | -179.95 (16) | C11A—C12A—C13A—C14A | -178 (3) |
| N2—C7—C8—C9 | 179.4 (2) | C11A—C12A—C13A—C15A | 10 (5) |

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> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| N3—H1N3...O1 ⁱ | 0.873 (17) | 2.052 (17) | 2.9167 (18) | 170.8 (16) |
| C4—H4A...O1 ⁱ | 0.93 | 2.53 | 3.251 (2) | 135 |

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