Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Indizoline[1]

**Hoong-Kun Fun,[a]*§ Wisanu Maneerat,[b] Surat Laphookhieo[b] and Suchada Chantrapromma[c]¶**

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]School of Science, Mae Fah Luang University, Tasud, Muang Chiang Rai 57100, Thailand, and [c]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

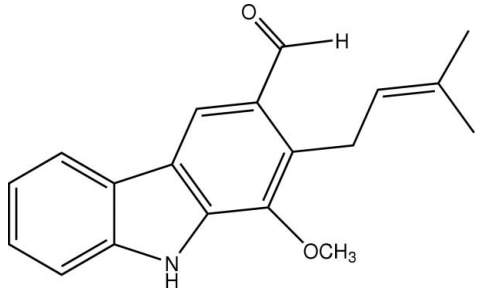
Received 9 September 2009; accepted 12 September 2009

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.042; $wR$ factor = 0.124; data-to-parameter ratio = 21.4.

The title compound [systematic name: 1-methoxy-2-(3-methylbut-2-enyl)-9*H*-carbazole-3-carbaldehyde], $C_{19}H_{19}NO_2$, is a natural carbazole which was isolated from the twigs of *Clausena lansium*. The carbazole ring system is essentially planar with a mean deviation of 0.0068 (10) Å. The aldehyde substituent is approximately co-planar with the attached benzene ring with a torsion angle of −8.58 (14)°, whereas the methoxy group is rotated out of the benzene plane with a torsion angle of −82.17 (11)°. The dihedral angle between the mean planes of the 3-methyl-2-butenyl group and the carbazole ring is 88.06 (5)°. An intermolecular N—H···O interaction connects the molecules into a chain along the *a* axis. The crystal is further consolidated by a C—H···O hydrogen bond and two $\pi$–$\pi$ interactions with centroid–centroid distances of 3.6592 (6) and 3.7440 (6) Å.

## Related literature

For bond-length data, see Allen *et al.* (1987). For background to carbazoles and their biological activity, see: Adebajo *et al.* (2009); Ito *et al.* (1998); Kumar *et al.* (1995); Lin (1989); Ng *et al.* (2003); Yang *et al.* (1988). For a related structure, see: Fun *et al.* (2007). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).




## Experimental

### Crystal data

$C_{19}H_{19}NO_2$
$M_r$ = 293.35
Triclinic, $P\overline{1}$
$a$ = 9.0467 (1) Å
$b$ = 9.3257 (1) Å
$c$ = 10.6927 (1) Å
$\alpha$ = 65.717 (1)°
$\beta$ = 86.994 (1)°
$\gamma$ = 68.323 (1)°
$V$ = 758.86 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 100 K
0.53 × 0.27 × 0.22 mm

### Data collection

Bruker APEXII CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.957, $T_{max}$ = 0.982
20515 measured reflections
4410 independent reflections
3887 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.124
$S$ = 1.05
4410 reflections
206 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*1···O2$^{i}$ | 0.825 (19) | 2.099 (19) | 2.8843 (13) | 158.8 (15) |
| C18—H18*A*···O1$^{ii}$ | 0.96 | 2.59 | 3.5369 (15) | 168 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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).

The authors thank Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012. SL thanks the Thailand Research Fund (grant No. RSA5280011) and Mae Fah Luang University for financial support. WM thanks Mae Fah Luang University for a PhD graduate student research grant.

[1] This paper is dedicated to Dato' Dr Chatar Singh, Foundation Dean, School of Physics, Universiti Sains Malaysia, whose 80th birthday falls on the 9th September 2009.
§ Thomson Reuters ResearcherID: A-3561-2009.
¶ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2459).

## References


Adebajo, A. C., Iwalewa, E. O., Obuotor, E. M., Ibikunle, G. F., Omisore, N. O., Adewunmi, C. O., Obaparusi, O. O., Klaes, M., Adetogun, G. E., Schmidt, T. J. & Verspohl, E. J. (2009). *J. Ethnopharmacol.* **122**, 10–19.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Fun, H.-K., Laphookhieo, S., Maneerat, W. & Chantrapromma, S. (2007). *Acta Cryst.* E**63**, o3964–o3965.

Ito, C., Katsuno, S. & Furukawa, H. (1998). *Chem. Pharm. Bull.* **46**, 341–343.

Kumar, V., Vallipuram, K., Adebajo, A. C. & Reisch, J. (1995). *Phytochemistry*, **40**, 1563–1565.

Lin, J. H. (1989). *Phytochemistry*, **28**, 621–622.

Ng, T. B., Lam, S. K. & Fong, W. P. (2003). *Biol. Chem.* **384**, 289–293.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Yang, M. H., Chen, Y. Y. & Huang, L. (1988). *Phytochemistry*, **27**, 445–450.

# supporting information

*Acta Cryst.* (2009). E**65**, o2497–o2498 [doi:10.1107/S1600536809036915]

## Indizoline

**Hoong-Kun Fun, Wisanu Maneerat, Surat Laphookhieo and Suchada Chantrapromma**

### S1. Comment

*Clausena lansium* (Wampee) belongs to the Rutaceae family. Several parts of this plant have been used as folk medicine in China and Taiwan, for example, the leaves have been used for the treatment of coughs, asthma and gastro-intestinal diseases and the seeds for gastro-intestinal diseases such as acute and chronic gastro-intestinal inflammation and ulcers (Adebajo *et al.*, 2009). In addition, the fruits are used for influenza, colds and abdominal colic pains in Philippines (Lin, 1989). In previous studies, a number of coumarins (Ito *et al.*, 1998; Kumar *et al.*, 1995) and alkaloids (Lin, 1989; Yang *et al.*, 1988) have been isolated from different parts of this plant. As part of our continuing study on the chemical constituents and bioactive compounds from Thai medicinal plants, we report herein the crystal structure of the title compound (I), which was isolated from the twigs of *Clausena lansium* were collected from Nan province in the northern part of Thailand.

In the structure of (I), $C_{19}H_{19}NO_2$ (Fig. 1), the carbazole ring system (C1–C12/N1) is essentially planar with a mean deviation of 0.0068 (10) Å. The aldehyde substituent is planarly attached to the benzene ring. The methoxy group is in an (+)-anti-clinal [torsion angle C19–O1–C11–C10 = 101.43 (10)°] whereas the 3-methyl-2-butenyl is in an (-)-*syn*-clinal [C9–C10–C14–C15 = -71.91 (11)°] conformation with respect to the attached benzene ring. The dihedral angle between the 3-methyl-2-butenyl moiety and the mean plane of carbazole ring is 88.06 (5)°. The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987) and are comparable to the related structure (Fun *et al.*, 2007).

In the crystal packing (Fig. 2), an N—H···O intermolecular interaction connects the molecules into one dimensional chains along the [1 0 0] direction. The crystal is further consolidated by C—H···O (Table 1) and $\pi\cdots\pi$ interactions with the $Cg_1\cdots Cg_2$ distance = 3.7440 (6) Å and $Cg_2\cdots Cg_3$ = 3.6592 (6) Å [symmetry code: (1 - *x*, 1 - *y*, -*z*) for both $Cg\cdots Cg$]. $Cg_1$, $Cg_2$ and $Cg_3$ are the centroids of C1–C6–C7–C12–N1, C1–C6 and C7–C12 rings, respectively.

### S2. Experimental

Twigs of *Clausena lansium* (6.73 kg) were successively extracted with $CH_2Cl_2$ and acetone, over the period of 3 days each at room temperature to provide the crude $CH_2Cl_2$ and acetone extracts, respectively. The $CH_2Cl_2$ and acetone extracts were combined (34.02 g) and then subjected to quick column chromatography over silica gel eluted by gradient of hexane-acetone (100% hexane to 100% acetone) giving seventeen fractions (A—Q). Fraction G (207.1 mg) was subjected to purification by column chromatography using 20% EtOAc-hexane to yield the title compound (27.1 mg). Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from $CH_2Cl_2$/acetone (1:1, *v*/*v*) after several days (*m.p.* 344–345 K).

### S3. Refinement

The H atom attached to N1 was located in a difference map and was isotropically refined. The remaining H atoms were placed in calculated positions with C—H = 0.93 Å for aromatic and CH, 0.97 for $CH_2$ and 0.96 Å for $CH_3$ atoms. The $U_{iso}$

values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.70 Å from C8 and the deepest hole is located at 0.91 Å from C11.

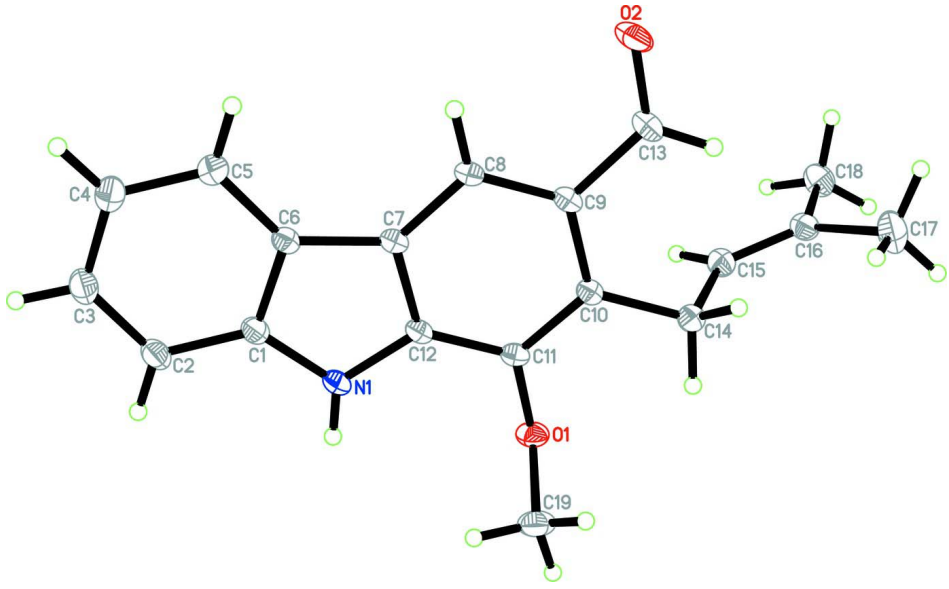



**Figure 1**

The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

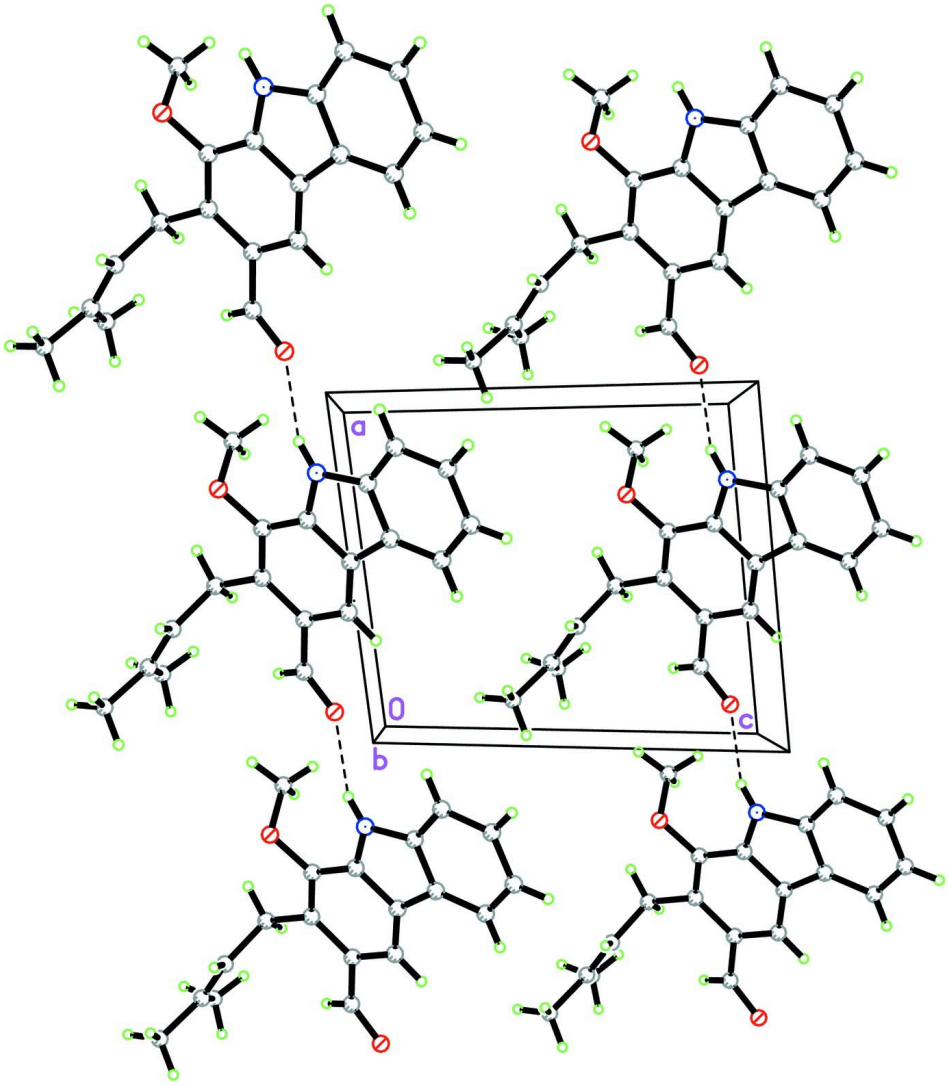



**Figure 2**

The crystal packing of (I) viewed along the *b* axis, showing one dimensional chains along the [1 0 0] direction. Hydrogen bonds are shown as dashed lines.

### 1-Methoxy-2-(3-methylbut-2-enyl)-9*H*-carbazole-3-carbaldehyde

*Crystal data*

$C_{19}H_{19}NO_2$
$M_r = 293.35$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.0467\ (1)$ Å
$b = 9.3257\ (1)$ Å
$c = 10.6927\ (1)$ Å
$\alpha = 65.717\ (1)°$
$\beta = 86.994\ (1)°$
$\gamma = 68.323\ (1)°$
$V = 758.86\ (2)$ Å$^3$
$Z = 2$
$F(000) = 312$
$D_x = 1.284$ Mg m$^{-3}$
Melting point = 344–345 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4410 reflections
$\theta = 2.1–30.0°$
$\mu = 0.08$ mm$^{-1}$
$T = 100$ K
Block, yellow
0.53 × 0.27 × 0.22 mm

*Data collection*

Bruker APEXII CCD area detector diffractometer
Radiation source: sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.957, $T_{max}$ = 0.982
20515 measured reflections
4410 independent reflections
3887 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 30.0°, $\theta_{min}$ = 2.1°
$h$ = −12→12
$k$ = −13→13
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.124
$S$ = 1.05
4410 reflections
206 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
$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2 + 0.2509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.26909 (8) | 0.78512 (9) | 0.28630 (7) | 0.01884 (16) |
| O2 | 0.90034 (9) | 0.78623 (10) | 0.07678 (8) | 0.02297 (17) |
| N1 | 0.23115 (10) | 0.73264 (11) | 0.03746 (9) | 0.01646 (17) |
| C1 | 0.26191 (11) | 0.70558 (11) | −0.08151 (10) | 0.01576 (18) |
| C2 | 0.16804 (12) | 0.67357 (12) | −0.15826 (11) | 0.0196 (2) |
| H2A | 0.0692 | 0.6690 | −0.1336 | 0.024* |
| C3 | 0.22843 (13) | 0.64886 (13) | −0.27314 (11) | 0.0212 (2) |
| H3A | 0.1685 | 0.6274 | −0.3264 | 0.025* |
| C4 | 0.37714 (13) | 0.65536 (13) | −0.31088 (10) | 0.0204 (2) |
| H4A | 0.4140 | 0.6386 | −0.3886 | 0.024* |
| C5 | 0.47039 (12) | 0.68656 (12) | −0.23362 (10) | 0.01765 (19) |
| H5A | 0.5694 | 0.6902 | −0.2585 | 0.021* |
| C6 | 0.41224 (11) | 0.71237 (11) | −0.11773 (9) | 0.01449 (17) |
| C7 | 0.47446 (11) | 0.74608 (11) | −0.01497 (9) | 0.01365 (17) |

| | | | | |
|---|---|---|---|---|
| C8 | 0.61476 (11) | 0.76411 (11) | 0.00817 (9) | 0.01443 (17) |
| H8A | 0.6938 | 0.7538 | −0.0505 | 0.017* |
| C9 | 0.63649 (11) | 0.79781 (11) | 0.11998 (9) | 0.01434 (17) |
| C10 | 0.51798 (11) | 0.81249 (11) | 0.21241 (9) | 0.01415 (17) |
| C11 | 0.38006 (11) | 0.78850 (11) | 0.19182 (9) | 0.01450 (18) |
| C12 | 0.35762 (11) | 0.75789 (11) | 0.07777 (9) | 0.01417 (17) |
| C13 | 0.78581 (11) | 0.81675 (12) | 0.14115 (10) | 0.01751 (19) |
| H13A | 0.7940 | 0.8543 | 0.2077 | 0.021* |
| C14 | 0.54154 (12) | 0.84670 (12) | 0.33525 (10) | 0.01731 (18) |
| H14A | 0.5751 | 0.9421 | 0.3046 | 0.021* |
| H14B | 0.4400 | 0.8784 | 0.3718 | 0.021* |
| C15 | 0.66415 (12) | 0.69530 (12) | 0.44873 (10) | 0.01723 (19) |
| H15A | 0.6619 | 0.5895 | 0.4679 | 0.021* |
| C16 | 0.77577 (12) | 0.69655 (12) | 0.52484 (10) | 0.01787 (19) |
| C17 | 0.79953 (15) | 0.85421 (14) | 0.50890 (13) | 0.0288 (2) |
| H17A | 0.7349 | 0.9490 | 0.4273 | 0.043* |
| H17B | 0.9103 | 0.8374 | 0.5010 | 0.043* |
| H17C | 0.7686 | 0.8769 | 0.5882 | 0.043* |
| C18 | 0.88710 (13) | 0.53395 (13) | 0.63786 (11) | 0.0229 (2) |
| H18A | 0.8557 | 0.4422 | 0.6485 | 0.034* |
| H18B | 0.8819 | 0.5463 | 0.7230 | 0.034* |
| H18C | 0.9947 | 0.5097 | 0.6141 | 0.034* |
| C19 | 0.13064 (13) | 0.94117 (15) | 0.24419 (12) | 0.0251 (2) |
| H19A | 0.0625 | 0.9333 | 0.3166 | 0.038* |
| H19B | 0.0730 | 0.9607 | 0.1621 | 0.038* |
| H19C | 0.1639 | 1.0339 | 0.2256 | 0.038* |
| H1N1 | 0.145 (2) | 0.7418 (19) | 0.0698 (16) | 0.029 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0144 (3) | 0.0228 (3) | 0.0189 (3) | −0.0072 (3) | 0.0064 (3) | −0.0089 (3) |
| O2 | 0.0127 (3) | 0.0318 (4) | 0.0239 (4) | −0.0103 (3) | 0.0028 (3) | −0.0097 (3) |
| N1 | 0.0107 (4) | 0.0217 (4) | 0.0199 (4) | −0.0077 (3) | 0.0032 (3) | −0.0102 (3) |
| C1 | 0.0129 (4) | 0.0159 (4) | 0.0182 (4) | −0.0051 (3) | 0.0004 (3) | −0.0070 (3) |
| C2 | 0.0148 (4) | 0.0203 (4) | 0.0247 (5) | −0.0065 (3) | −0.0012 (3) | −0.0103 (4) |
| C3 | 0.0208 (5) | 0.0206 (4) | 0.0226 (5) | −0.0067 (4) | −0.0033 (4) | −0.0100 (4) |
| C4 | 0.0237 (5) | 0.0197 (4) | 0.0182 (4) | −0.0073 (4) | 0.0003 (4) | −0.0090 (3) |
| C5 | 0.0175 (4) | 0.0177 (4) | 0.0176 (4) | −0.0064 (3) | 0.0026 (3) | −0.0077 (3) |
| C6 | 0.0123 (4) | 0.0139 (4) | 0.0161 (4) | −0.0043 (3) | 0.0005 (3) | −0.0056 (3) |
| C7 | 0.0108 (4) | 0.0138 (4) | 0.0153 (4) | −0.0045 (3) | 0.0014 (3) | −0.0052 (3) |
| C8 | 0.0105 (4) | 0.0158 (4) | 0.0160 (4) | −0.0050 (3) | 0.0027 (3) | −0.0060 (3) |
| C9 | 0.0107 (4) | 0.0147 (4) | 0.0163 (4) | −0.0051 (3) | 0.0006 (3) | −0.0050 (3) |
| C10 | 0.0120 (4) | 0.0142 (4) | 0.0153 (4) | −0.0047 (3) | 0.0007 (3) | −0.0055 (3) |
| C11 | 0.0112 (4) | 0.0160 (4) | 0.0160 (4) | −0.0054 (3) | 0.0034 (3) | −0.0064 (3) |
| C12 | 0.0102 (4) | 0.0150 (4) | 0.0166 (4) | −0.0048 (3) | 0.0011 (3) | −0.0060 (3) |
| C13 | 0.0133 (4) | 0.0201 (4) | 0.0178 (4) | −0.0077 (3) | −0.0002 (3) | −0.0052 (3) |
| C14 | 0.0152 (4) | 0.0192 (4) | 0.0179 (4) | −0.0052 (3) | 0.0007 (3) | −0.0093 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0171 (4) | 0.0177 (4) | 0.0166 (4) | −0.0071 (3) | 0.0021 (3) | −0.0066 (3) |
| C16 | 0.0156 (4) | 0.0200 (4) | 0.0173 (4) | −0.0065 (3) | 0.0017 (3) | −0.0075 (3) |
| C17 | 0.0303 (6) | 0.0248 (5) | 0.0321 (6) | −0.0115 (4) | −0.0063 (4) | −0.0109 (4) |
| C18 | 0.0216 (5) | 0.0231 (5) | 0.0200 (4) | −0.0078 (4) | −0.0023 (4) | −0.0053 (4) |
| C19 | 0.0161 (5) | 0.0297 (5) | 0.0280 (5) | −0.0041 (4) | 0.0061 (4) | −0.0152 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C11 | 1.3857 (11) | C9—C13 | 1.4658 (13) |
| O1—C19 | 1.4363 (13) | C10—C11 | 1.3895 (12) |
| O2—C13 | 1.2235 (12) | C10—C14 | 1.5155 (12) |
| N1—C12 | 1.3727 (11) | C11—C12 | 1.4018 (12) |
| N1—C1 | 1.3909 (12) | C13—H13A | 0.9300 |
| N1—H1N1 | 0.822 (17) | C14—C15 | 1.5071 (13) |
| C1—C2 | 1.3948 (13) | C14—H14A | 0.9700 |
| C1—C6 | 1.4113 (13) | C14—H14B | 0.9700 |
| C2—C3 | 1.3892 (14) | C15—C16 | 1.3360 (13) |
| C2—H2A | 0.9300 | C15—H15A | 0.9300 |
| C3—C4 | 1.3999 (15) | C16—C17 | 1.5035 (14) |
| C3—H3A | 0.9300 | C16—C18 | 1.5054 (14) |
| C4—C5 | 1.3895 (14) | C17—H17A | 0.9600 |
| C4—H4A | 0.9300 | C17—H17B | 0.9600 |
| C5—C6 | 1.3978 (13) | C17—H17C | 0.9600 |
| C5—H5A | 0.9300 | C18—H18A | 0.9600 |
| C6—C7 | 1.4500 (12) | C18—H18B | 0.9600 |
| C7—C8 | 1.3875 (12) | C18—H18C | 0.9600 |
| C7—C12 | 1.4167 (12) | C19—H19A | 0.9600 |
| C8—C9 | 1.3951 (13) | C19—H19B | 0.9600 |
| C8—H8A | 0.9300 | C19—H19C | 0.9600 |
| C9—C10 | 1.4271 (13) | | |

| | | | |
|---|---|---|---|
| C11—O1—C19 | 113.68 (8) | N1—C12—C11 | 128.96 (9) |
| C12—N1—C1 | 108.62 (8) | N1—C12—C7 | 109.54 (8) |
| C12—N1—H1N1 | 128.2 (11) | C11—C12—C7 | 121.50 (8) |
| C1—N1—H1N1 | 122.8 (11) | O2—C13—C9 | 124.13 (9) |
| N1—C1—C2 | 128.87 (9) | O2—C13—H13A | 117.9 |
| N1—C1—C6 | 109.20 (8) | C9—C13—H13A | 117.9 |
| C2—C1—C6 | 121.92 (9) | C15—C14—C10 | 112.82 (8) |
| C3—C2—C1 | 117.16 (9) | C15—C14—H14A | 109.0 |
| C3—C2—H2A | 121.4 | C10—C14—H14A | 109.0 |
| C1—C2—H2A | 121.4 | C15—C14—H14B | 109.0 |
| C2—C3—C4 | 121.77 (9) | C10—C14—H14B | 109.0 |
| C2—C3—H3A | 119.1 | H14A—C14—H14B | 107.8 |
| C4—C3—H3A | 119.1 | C16—C15—C14 | 127.08 (9) |
| C5—C4—C3 | 120.78 (9) | C16—C15—H15A | 116.5 |
| C5—C4—H4A | 119.6 | C14—C15—H15A | 116.5 |
| C3—C4—H4A | 119.6 | C15—C16—C17 | 124.45 (9) |
| C4—C5—C6 | 118.63 (9) | C15—C16—C18 | 120.70 (9) |

| | | | |
|---|---|---|---|
| C4—C5—H5A | 120.7 | C17—C16—C18 | 114.84 (9) |
| C6—C5—H5A | 120.7 | C16—C17—H17A | 109.5 |
| C5—C6—C1 | 119.73 (9) | C16—C17—H17B | 109.5 |
| C5—C6—C7 | 133.90 (9) | H17A—C17—H17B | 109.5 |
| C1—C6—C7 | 106.36 (8) | C16—C17—H17C | 109.5 |
| C8—C7—C12 | 119.22 (8) | H17A—C17—H17C | 109.5 |
| C8—C7—C6 | 134.49 (8) | H17B—C17—H17C | 109.5 |
| C12—C7—C6 | 106.27 (8) | C16—C18—H18A | 109.5 |
| C7—C8—C9 | 119.55 (8) | C16—C18—H18B | 109.5 |
| C7—C8—H8A | 120.2 | H18A—C18—H18B | 109.5 |
| C9—C8—H8A | 120.2 | C16—C18—H18C | 109.5 |
| C8—C9—C10 | 121.40 (8) | H18A—C18—H18C | 109.5 |
| C8—C9—C13 | 118.14 (8) | H18B—C18—H18C | 109.5 |
| C10—C9—C13 | 120.46 (8) | O1—C19—H19A | 109.5 |
| C11—C10—C9 | 118.94 (8) | O1—C19—H19B | 109.5 |
| C11—C10—C14 | 119.26 (8) | H19A—C19—H19B | 109.5 |
| C9—C10—C14 | 121.76 (8) | O1—C19—H19C | 109.5 |
| O1—C11—C10 | 120.95 (8) | H19A—C19—H19C | 109.5 |
| O1—C11—C12 | 119.63 (8) | H19B—C19—H19C | 109.5 |
| C10—C11—C12 | 119.33 (8) | | |

| | | | |
|---|---|---|---|
| C12—N1—C1—C2 | 179.57 (9) | C13—C9—C10—C14 | −0.39 (13) |
| C12—N1—C1—C6 | 0.45 (10) | C19—O1—C11—C10 | 101.43 (10) |
| N1—C1—C2—C3 | −179.21 (9) | C19—O1—C11—C12 | −82.17 (11) |
| C6—C1—C2—C3 | −0.19 (14) | C9—C10—C11—O1 | 173.69 (8) |
| C1—C2—C3—C4 | 0.09 (15) | C14—C10—C11—O1 | −3.97 (13) |
| C2—C3—C4—C5 | 0.20 (15) | C9—C10—C11—C12 | −2.73 (13) |
| C3—C4—C5—C6 | −0.37 (14) | C14—C10—C11—C12 | 179.61 (8) |
| C4—C5—C6—C1 | 0.27 (14) | C1—N1—C12—C11 | −179.49 (9) |
| C4—C5—C6—C7 | 179.54 (9) | C1—N1—C12—C7 | −0.48 (10) |
| N1—C1—C6—C5 | 179.20 (8) | O1—C11—C12—N1 | 4.24 (15) |
| C2—C1—C6—C5 | 0.01 (14) | C10—C11—C12—N1 | −179.30 (9) |
| N1—C1—C6—C7 | −0.25 (10) | O1—C11—C12—C7 | −174.67 (8) |
| C2—C1—C6—C7 | −179.44 (8) | C10—C11—C12—C7 | 1.79 (14) |
| C5—C6—C7—C8 | −0.63 (18) | C8—C7—C12—N1 | −178.66 (8) |
| C1—C6—C7—C8 | 178.71 (10) | C6—C7—C12—N1 | 0.31 (10) |
| C5—C6—C7—C12 | −179.38 (10) | C8—C7—C12—C11 | 0.44 (13) |
| C1—C6—C7—C12 | −0.04 (10) | C6—C7—C12—C11 | 179.42 (8) |
| C12—C7—C8—C9 | −1.64 (13) | C8—C9—C13—O2 | −8.58 (14) |
| C6—C7—C8—C9 | 179.73 (9) | C10—C9—C13—O2 | 170.98 (9) |
| C7—C8—C9—C10 | 0.67 (13) | C11—C10—C14—C15 | 105.69 (10) |
| C7—C8—C9—C13 | −179.77 (8) | C9—C10—C14—C15 | −71.91 (11) |
| C8—C9—C10—C11 | 1.55 (13) | C10—C14—C15—C16 | 139.14 (10) |
| C13—C9—C10—C11 | −177.99 (8) | C14—C15—C16—C17 | −0.51 (17) |
| C8—C9—C10—C14 | 179.15 (8) | C14—C15—C16—C18 | 178.19 (9) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*1···O2$^{i}$ | 0.825 (19) | 2.099 (19) | 2.8843 (13) | 158.8 (15) |
| C18—H18*A*···O1$^{ii}$ | 0.96 | 2.59 | 3.5369 (15) | 168 |

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