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

# 5-Methoxy-1-[(5-methoxy-1*H*-indol-2-yl)methyl]-1*H*-indole

**Mohamed I. Attia,[a] Nasser R. El-Brollosy,[a] Hazem A. Ghabbour,[a] Suhana Arshad[b] and Hoong-Kun Fun[b]*‡**

[a]Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, Riyadh 11451, Saudi Arabia, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

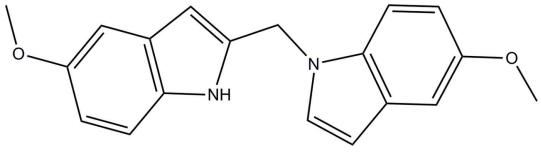
Received 29 February 2012; accepted 1 March 2012

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.046; $wR$ factor = 0.133; data-to-parameter ratio = 12.0.

In the title compound, $C_{19}H_{18}N_2O_2$, the two indole ring systems are essentially planar [maximum deviation = 0.015 (2) Å in both indole ring systems] and make a dihedral angle of 72.17 (7)° with each other. In the crystal, the molecules are linked into a zigzag chain along the *a* axis *via* N—H···O hydrogen bonds.

## Related literature

For the biological activity of melatonin (MLT), see: Csernus & Mess (2003); Nosjean *et al.* (2000); Blask *et al.* (2002); Genovese *et al.* (2005); Mills *et al.* (2005); Peres (2005); Sofic *et al.* (2005); Witt-Enderby *et al.* (2006). For related structures, see: Narayanan *et al.* (2011); Deng *et al.* (2011). For the synthesis, see: Attia *et al.* (2008).




## Experimental

### Crystal data

$C_{19}H_{18}N_2O_2$
$M_r$ = 306.35
Monoclinic, $P2_1/c$
$a$ = 9.4446 (5) Å
$b$ = 19.5625 (8) Å
$c$ = 8.6657 (5) Å
$\beta$ = 98.903 (4)°
$V$ = 1581.78 (14) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.68 mm$^{-1}$
$T$ = 296 K
0.92 × 0.20 × 0.06 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.575, $T_{max}$ = 0.961
9421 measured reflections
2584 independent reflections
2087 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.133
$S$ = 1.06
2584 reflections
215 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H1*N2*···O1$^{i}$ | 0.88 (2) | 2.24 (3) | 3.037 (2) | 151 (2) |

Symmetry code: (i) $x-1, y, z$.

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).

The authors thank the Deanship of Scientific Research and the Research Center of the College of Pharmacy, King Saud University, for supporting this study. HKF and SA thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). SA also thanks the Malaysian Government and USM for the Academic Staff Training Scheme (ASTS) award.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5084).

## References

Attia, M. I., Witt-Enderby, P. A. & Julius, J. (2008). *Bioorg. Med. Chem.* **16**, 7654–7661.
Blask, D. E., Sauer, L. A. & Dauchy, R. T. (2002). *Curr. Top. Med. Chem.* **2**, 113–132.
Bruker (2009). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Csernus, V. & Mess, B. (2003). *Neuroendocrinol. Lett.* **24**, 404–411.
Deng, X., Wu, D., Huang, X. & Luo, F. (2011). *Acta Cryst.* E**67**, o1603.
Genovese, T., Mazzon, E., Muia, C., Bramanti, P., De Sarro, A. & Cuzzocrea, S. (2005). *J. Pineal Res.* **38**, 198–208.
Mills, E., Wu, P., Seely, D. & Guyatt, G. (2005). *J. Pineal Res.* **39**, 360–366.
Narayanan, P., Sethusankar, K., Ramachandiran, K. & Perumal, P. T. (2011). *Acta Cryst.* E**67**, o3196.
Nosjean, O., Ferro, M., Coge, F., Beauverger, P., Henlin, J.-M., Lefoulon, F., Fauchere, J. L., Delagrange, P., Canet, E. & Boutin, J. A. (2000). *J. Biol. Chem.* **275**, 31311–31317.
Peres, M. F. P. (2005). *Cephalalgia*, **5**, 403–411.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sofic, E., Rimpapa, Z., Kundurovic, Z., Sapcanin, A., Tahirovic, I., Rustembegovic, A. & Cao, G. (2005). *J. Neural Transm.* **112**, 349–358.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Witt-Enderby, P. A., Radio, N. M., Doctor, J. S. & Davis, V. L. (2006). *J. Pineal Res.* **41**, 297–305.

‡ Thomson Reuters ResearcherID: A-3561-2009.

# supplementary materials

*Acta Cryst.* (2012). E**68**, o971 [doi:10.1107/S1600536812009257]

## 5-Methoxy-1-[(5-methoxy-1*H*-indol-2-yl)methyl]-1*H*-indole

**Mohamed I. Attia, Nasser R. El-Brollosy, Hazem A. Ghabbour, Suhana Arshad and Hoong-Kun Fun**

### Comment

Melatonin (*N*-acetyl-5-methoxytryptamine, MLT) is primarily produced by the pineal gland in the brain with a marked circadian rhythm normally peaking in the dark to regulate sleep. MLT acts through activation of two G-protein-coupled receptors, designated as $MT_1$ and $MT_2$ (Csernus & Mess, 2003). In addition, a low-affinity putative MLT binding site called $MT_3$ has been recently characterized as a melatonin-sensitive form of the human enzyme quinine reductase 2 (Nosjean *et al.*, 2000). MLT has found widespread use in the treatment of sleep disorders. Other effects described in the literature include its anti-inflammatory, pain modulatory, antitumor, and antioxidant properties (Blask *et al.*, 2002; Genovese *et al.*, 2005; Mills *et al.*, 2005; Peres, 2005; Sofic *et al.*, 2005; Witt-Enderby *et al.*, 2006). The title compound is an intermediate which could yield, *via* the reported procedure (Attia *et al.*, 2008), various MLT analogues which can be evaluated for their potency and selectivity for MLT receptor subtypes.

In the title compound (Fig. 1), the indole ring systems (N1/C10–C17 & N2/C1–C8) are essentially planar with maximum deviations of 0.015 (2) Å at atom C10 and C2, respectively. In addition, the indole ring systems are almost perpendicular to each other with dihedral angle of 72.17 (7)°. Bond lengths and angles are within the normal range and are comparable to those in the related structures (Narayanan *et al.*, 2011; Deng *et al.*, 2011).

The crystal structure is shown in Fig. 2. The molecules are linked into one dimensional zigzag chains along *a*-axis *via* N2—H1N2···O1 interactions (Table 1).

### Experimental

(5-Methoxy-1*H*-indol-1-yl)(5-methoxy-1*H*-indol-2-yl)methanone (0.50 g, 156.03 mmol) was dissolved in dry THF (5 ml) and was added drop-wise to a cooled (0 °C) suspension of $LiAlH_4/AlCl_3$ in dry diethyl ether (prepared by a slow addition of $AlCl_3$ (0.32 g, 2.41 mmol) to a suspension $LiAlH_4$ (0.27 g, 7.13 mmol) in dry diethyl ether (15 ml) at 0 °C). The resulting reaction mixture was stirred at 0 °C for one hour and at room temperature for another one hour. The reaction was quenched by a slow addition of saturated sodium sulfate solution. The solids formed were removed by filtration, washed with chloroform (20 ml) and the combined organic phase was dried ($Na_2SO_4$) and evaporated under reduced pressure. The residue was purified by silica gel chromatography (chloroform/methanol/ammonia, 10.0:1.0:0.1) to produce the title compound as a light red powder which was recrystallized from ethanol to give single crystals (*m.p.* 173–174 °C).

### Refinement

N-bound H atom was located in a difference Fourier map and refined freely [N—H = 0.88 (2) Å]. Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C). A rotating group model was applied to the methyl groups.

### Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (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).

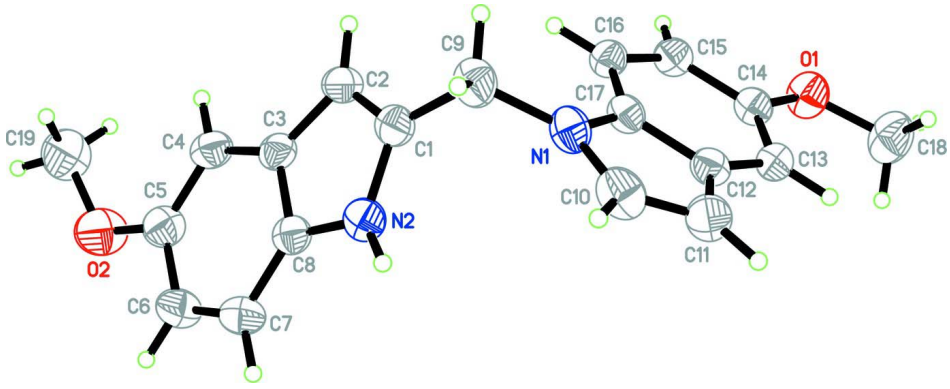



**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

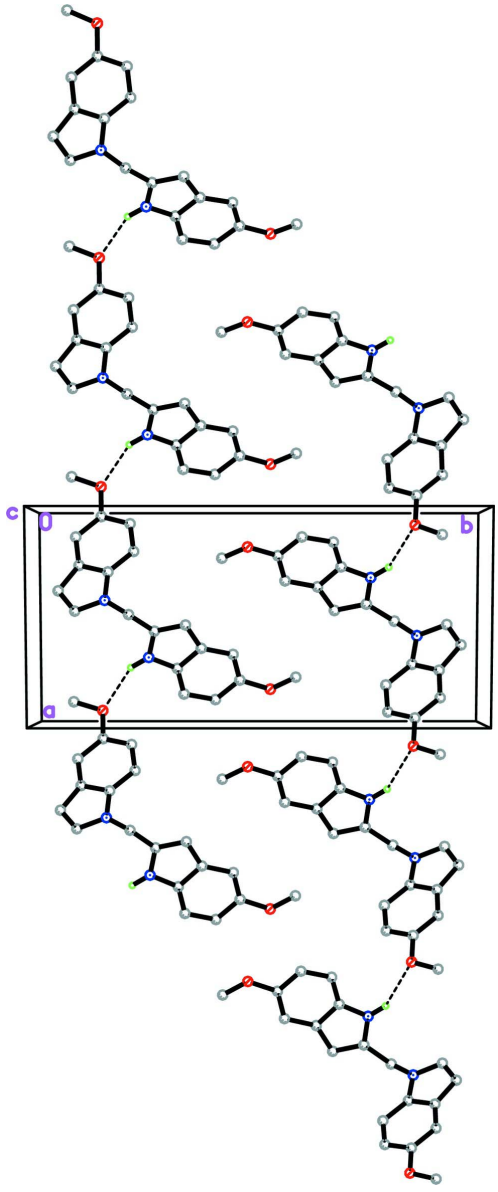



**Figure 2**

A packing diagram of the title compound viewed along the *c* axis. For the sake of clarity, H atoms not involved in the intermolecular interactions (dashed lines) have been omitted.

### 5-Methoxy-1-[(5-methoxy-1*H*-indol-2-yl)methyl]-1*H*-indole

*Crystal data*

$C_{19}H_{18}N_2O_2$
$M_r = 306.35$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.4446$ (5) Å
$b = 19.5625$ (8) Å
$c = 8.6657$ (5) Å
$\beta = 98.903$ (4)°
$V = 1581.78$ (14) Å$^3$
$Z = 4$
$F(000) = 648$
$D_x = 1.286$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 919 reflections
$\theta = 4.5$–60.8°
$\mu = 0.68$ mm$^{-1}$

$T$ = 296 K
Plate, pink

0.92 × 0.20 × 0.06 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.575, $T_{max}$ = 0.961

9421 measured reflections
2584 independent reflections
2087 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041
$\theta_{max}$ = 65.0°, $\theta_{min}$ = 4.5°
$h$ = −11→11
$k$ = −22→22
$l$ = −8→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.133
$S$ = 1.06
2584 reflections
215 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
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.3465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (5)

*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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.57956 (17) | 0.66569 (8) | 0.6878 (2) | 0.0604 (4) |
| N2 | 0.32036 (18) | 0.76234 (9) | 0.5805 (2) | 0.0588 (4) |
| O1 | 1.08253 (15) | 0.66541 (8) | 0.44352 (19) | 0.0727 (5) |
| O2 | 0.1802 (2) | 1.02435 (9) | 0.3902 (2) | 0.0978 (6) |
| C1 | 0.4392 (2) | 0.77296 (11) | 0.6916 (2) | 0.0573 (5) |
| C2 | 0.4672 (2) | 0.84059 (11) | 0.7021 (2) | 0.0616 (5) |
| H2A | 0.5421 | 0.8610 | 0.7683 | 0.074* |
| C3 | 0.36179 (19) | 0.87542 (10) | 0.5938 (2) | 0.0536 (5) |
| C4 | 0.3345 (2) | 0.94428 (11) | 0.5531 (3) | 0.0644 (6) |
| H4A | 0.3924 | 0.9789 | 0.6018 | 0.077* |
| C5 | 0.2203 (2) | 0.95918 (11) | 0.4396 (3) | 0.0675 (6) |
| C6 | 0.1345 (2) | 0.90736 (12) | 0.3641 (3) | 0.0703 (6) |
| H6A | 0.0593 | 0.9188 | 0.2859 | 0.084* |

| | | | | |
|---|---|---|---|---|
| C7 | 0.1585 (2) | 0.84013 (12) | 0.4025 (3) | 0.0661 (6) |
| H7A | 0.1008 | 0.8059 | 0.3519 | 0.079* |
| C8 | 0.27154 (19) | 0.82467 (9) | 0.5191 (2) | 0.0529 (5) |
| C9 | 0.5147 (2) | 0.71548 (12) | 0.7821 (3) | 0.0709 (6) |
| H9A | 0.5893 | 0.7342 | 0.8602 | 0.085* |
| H9B | 0.4470 | 0.6918 | 0.8368 | 0.085* |
| C10 | 0.5303 (2) | 0.60163 (11) | 0.6481 (3) | 0.0728 (6) |
| H10A | 0.4461 | 0.5831 | 0.6732 | 0.087* |
| C11 | 0.6216 (2) | 0.56865 (11) | 0.5667 (3) | 0.0704 (6) |
| H11A | 0.6112 | 0.5244 | 0.5271 | 0.084* |
| C12 | 0.73578 (19) | 0.61433 (9) | 0.5536 (2) | 0.0535 (5) |
| C13 | 0.86132 (19) | 0.60976 (9) | 0.4852 (2) | 0.0552 (5) |
| H13A | 0.8813 | 0.5708 | 0.4311 | 0.066* |
| C14 | 0.95290 (19) | 0.66412 (10) | 0.5005 (2) | 0.0538 (5) |
| C15 | 0.9232 (2) | 0.72402 (10) | 0.5783 (2) | 0.0565 (5) |
| H15A | 0.9874 | 0.7603 | 0.5852 | 0.068* |
| C16 | 0.8003 (2) | 0.72991 (9) | 0.6444 (2) | 0.0548 (5) |
| H16A | 0.7802 | 0.7696 | 0.6961 | 0.066* |
| C17 | 0.70772 (19) | 0.67448 (9) | 0.6312 (2) | 0.0505 (4) |
| C18 | 1.1240 (3) | 0.60545 (15) | 0.3717 (4) | 0.0934 (8) |
| H18A | 1.2164 | 0.6122 | 0.3411 | 0.140* |
| H18B | 1.0550 | 0.5953 | 0.2811 | 0.140* |
| H18C | 1.1287 | 0.5680 | 0.4440 | 0.140* |
| C19 | 0.2324 (3) | 1.07922 (13) | 0.4857 (4) | 0.1022 (9) |
| H19A | 0.1819 | 1.1201 | 0.4492 | 0.153* |
| H19C | 0.3328 | 1.0851 | 0.4823 | 0.153* |
| H19D | 0.2184 | 1.0701 | 0.5911 | 0.153* |
| H1N2 | 0.274 (3) | 0.7238 (12) | 0.559 (3) | 0.072 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0524 (9) | 0.0644 (10) | 0.0664 (11) | 0.0041 (7) | 0.0159 (7) | 0.0093 (8) |
| N2 | 0.0544 (9) | 0.0598 (10) | 0.0637 (11) | −0.0052 (8) | 0.0135 (7) | 0.0005 (8) |
| O1 | 0.0617 (8) | 0.0756 (10) | 0.0863 (11) | −0.0066 (7) | 0.0287 (7) | −0.0043 (8) |
| O2 | 0.1246 (15) | 0.0672 (10) | 0.0933 (14) | 0.0037 (9) | −0.0091 (11) | 0.0129 (9) |
| C1 | 0.0501 (10) | 0.0742 (13) | 0.0501 (12) | 0.0041 (9) | 0.0153 (8) | −0.0015 (9) |
| C2 | 0.0528 (11) | 0.0777 (14) | 0.0531 (12) | −0.0001 (9) | 0.0040 (8) | −0.0115 (9) |
| C3 | 0.0514 (10) | 0.0635 (11) | 0.0475 (11) | −0.0020 (8) | 0.0132 (8) | −0.0084 (8) |
| C4 | 0.0669 (12) | 0.0638 (13) | 0.0615 (13) | −0.0084 (10) | 0.0073 (10) | −0.0102 (9) |
| C5 | 0.0758 (13) | 0.0634 (12) | 0.0624 (14) | 0.0007 (10) | 0.0078 (10) | 0.0034 (10) |
| C6 | 0.0682 (13) | 0.0769 (14) | 0.0617 (14) | −0.0004 (11) | −0.0031 (10) | 0.0038 (10) |
| C7 | 0.0601 (12) | 0.0718 (14) | 0.0634 (13) | −0.0117 (10) | 0.0005 (10) | −0.0038 (10) |
| C8 | 0.0495 (10) | 0.0603 (11) | 0.0513 (11) | −0.0038 (8) | 0.0147 (8) | −0.0033 (8) |
| C9 | 0.0676 (13) | 0.0903 (16) | 0.0584 (14) | 0.0158 (11) | 0.0217 (10) | 0.0089 (11) |
| C10 | 0.0542 (11) | 0.0711 (14) | 0.0938 (18) | −0.0107 (10) | 0.0132 (11) | 0.0141 (12) |
| C11 | 0.0587 (12) | 0.0594 (12) | 0.0917 (17) | −0.0093 (10) | 0.0079 (11) | −0.0037 (11) |
| C12 | 0.0482 (10) | 0.0538 (10) | 0.0563 (12) | −0.0028 (8) | 0.0015 (8) | 0.0027 (8) |
| C13 | 0.0543 (10) | 0.0534 (10) | 0.0569 (12) | 0.0020 (8) | 0.0061 (8) | −0.0050 (8) |
| C14 | 0.0486 (10) | 0.0600 (11) | 0.0534 (11) | −0.0017 (8) | 0.0097 (8) | 0.0042 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0546 (10) | 0.0527 (11) | 0.0618 (13) | −0.0058 (8) | 0.0078 (8) | 0.0018 (8) |
| C16 | 0.0592 (11) | 0.0502 (10) | 0.0538 (12) | 0.0011 (8) | 0.0053 (8) | −0.0016 (8) |
| C17 | 0.0479 (9) | 0.0530 (10) | 0.0502 (10) | 0.0031 (8) | 0.0064 (7) | 0.0072 (8) |
| C18 | 0.0780 (16) | 0.1011 (19) | 0.110 (2) | 0.0006 (14) | 0.0436 (14) | −0.0225 (15) |
| C19 | 0.112 (2) | 0.0623 (15) | 0.128 (3) | 0.0008 (14) | 0.0042 (18) | 0.0030 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C10 | 1.362 (3) | C7—H7A | 0.9300 |
| N1—C17 | 1.385 (2) | C9—H9A | 0.9700 |
| N1—C9 | 1.465 (3) | C9—H9B | 0.9700 |
| N2—C1 | 1.377 (3) | C10—C11 | 1.359 (3) |
| N2—C8 | 1.381 (3) | C10—H10A | 0.9300 |
| N2—H1N2 | 0.88 (2) | C11—C12 | 1.419 (3) |
| O1—C14 | 1.390 (2) | C11—H11A | 0.9300 |
| O1—C18 | 1.411 (3) | C12—C17 | 1.401 (3) |
| O2—C5 | 1.379 (3) | C12—C13 | 1.408 (3) |
| O2—C19 | 1.398 (3) | C13—C14 | 1.364 (3) |
| C1—C2 | 1.349 (3) | C13—H13A | 0.9300 |
| C1—C9 | 1.488 (3) | C14—C15 | 1.402 (3) |
| C2—C3 | 1.431 (3) | C15—C16 | 1.376 (3) |
| C2—H2A | 0.9300 | C15—H15A | 0.9300 |
| C3—C8 | 1.400 (3) | C16—C17 | 1.387 (3) |
| C3—C4 | 1.406 (3) | C16—H16A | 0.9300 |
| C4—C5 | 1.374 (3) | C18—H18A | 0.9600 |
| C4—H4A | 0.9300 | C18—H18B | 0.9600 |
| C5—C6 | 1.396 (3) | C18—H18C | 0.9600 |
| C6—C7 | 1.367 (3) | C19—H19A | 0.9600 |
| C6—H6A | 0.9300 | C19—H19C | 0.9600 |
| C7—C8 | 1.385 (3) | C19—H19D | 0.9600 |
| | | | |
| C10—N1—C17 | 107.95 (17) | H9A—C9—H9B | 107.6 |
| C10—N1—C9 | 126.61 (18) | C11—C10—N1 | 110.38 (18) |
| C17—N1—C9 | 125.33 (17) | C11—C10—H10A | 124.8 |
| C1—N2—C8 | 108.88 (17) | N1—C10—H10A | 124.8 |
| C1—N2—H1N2 | 127.1 (16) | C10—C11—C12 | 107.10 (19) |
| C8—N2—H1N2 | 123.6 (16) | C10—C11—H11A | 126.5 |
| C14—O1—C18 | 117.49 (17) | C12—C11—H11A | 126.5 |
| C5—O2—C19 | 118.1 (2) | C17—C12—C13 | 119.31 (16) |
| C2—C1—N2 | 108.97 (18) | C17—C12—C11 | 106.74 (18) |
| C2—C1—C9 | 129.2 (2) | C13—C12—C11 | 133.94 (19) |
| N2—C1—C9 | 121.79 (19) | C14—C13—C12 | 118.18 (17) |
| C1—C2—C3 | 108.28 (17) | C14—C13—H13A | 120.9 |
| C1—C2—H2A | 125.9 | C12—C13—H13A | 120.9 |
| C3—C2—H2A | 125.9 | C13—C14—O1 | 124.04 (18) |
| C8—C3—C4 | 119.24 (18) | C13—C14—C15 | 121.81 (18) |
| C8—C3—C2 | 106.12 (17) | O1—C14—C15 | 114.14 (16) |
| C4—C3—C2 | 134.64 (18) | C16—C15—C14 | 120.99 (17) |
| C5—C4—C3 | 118.42 (19) | C16—C15—H15A | 119.5 |
| C5—C4—H4A | 120.8 | C14—C15—H15A | 119.5 |

| | | | |
|---|---|---|---|
| C3—C4—H4A | 120.8 | C15—C16—C17 | 117.50 (17) |
| C4—C5—O2 | 124.4 (2) | C15—C16—H16A | 121.2 |
| C4—C5—C6 | 121.1 (2) | C17—C16—H16A | 121.2 |
| O2—C5—C6 | 114.5 (2) | N1—C17—C16 | 129.98 (18) |
| C7—C6—C5 | 121.5 (2) | N1—C17—C12 | 107.83 (16) |
| C7—C6—H6A | 119.3 | C16—C17—C12 | 122.19 (18) |
| C5—C6—H6A | 119.3 | O1—C18—H18A | 109.5 |
| C6—C7—C8 | 117.88 (19) | O1—C18—H18B | 109.5 |
| C6—C7—H7A | 121.1 | H18A—C18—H18B | 109.5 |
| C8—C7—H7A | 121.1 | O1—C18—H18C | 109.5 |
| N2—C8—C7 | 130.36 (18) | H18A—C18—H18C | 109.5 |
| N2—C8—C3 | 107.76 (17) | H18B—C18—H18C | 109.5 |
| C7—C8—C3 | 121.86 (19) | O2—C19—H19A | 109.5 |
| N1—C9—C1 | 114.58 (18) | O2—C19—H19C | 109.5 |
| N1—C9—H9A | 108.6 | H19A—C19—H19C | 109.5 |
| C1—C9—H9A | 108.6 | O2—C19—H19D | 109.5 |
| N1—C9—H9B | 108.6 | H19A—C19—H19D | 109.5 |
| C1—C9—H9B | 108.6 | H19C—C19—H19D | 109.5 |

| | | | |
|---|---|---|---|
| C8—N2—C1—C2 | −0.1 (2) | N2—C1—C9—N1 | 63.6 (3) |
| C8—N2—C1—C9 | 178.52 (17) | C17—N1—C10—C11 | −0.4 (2) |
| N2—C1—C2—C3 | 0.3 (2) | C9—N1—C10—C11 | −176.8 (2) |
| C9—C1—C2—C3 | −178.2 (2) | N1—C10—C11—C12 | 0.0 (3) |
| C1—C2—C3—C8 | −0.3 (2) | C10—C11—C12—C17 | 0.5 (2) |
| C1—C2—C3—C4 | 179.3 (2) | C10—C11—C12—C13 | 179.3 (2) |
| C8—C3—C4—C5 | −0.7 (3) | C17—C12—C13—C14 | 1.4 (3) |
| C2—C3—C4—C5 | 179.7 (2) | C11—C12—C13—C14 | −177.2 (2) |
| C3—C4—C5—O2 | 179.0 (2) | C12—C13—C14—O1 | 177.71 (17) |
| C3—C4—C5—C6 | −1.1 (3) | C12—C13—C14—C15 | −1.6 (3) |
| C19—O2—C5—C4 | −17.5 (4) | C18—O1—C14—C13 | −2.9 (3) |
| C19—O2—C5—C6 | 162.6 (2) | C18—O1—C14—C15 | 176.4 (2) |
| C4—C5—C6—C7 | 1.6 (4) | C13—C14—C15—C16 | 0.9 (3) |
| O2—C5—C6—C7 | −178.5 (2) | O1—C14—C15—C16 | −178.47 (17) |
| C5—C6—C7—C8 | −0.2 (3) | C14—C15—C16—C17 | 0.0 (3) |
| C1—N2—C8—C7 | 178.2 (2) | C10—N1—C17—C16 | −178.4 (2) |
| C1—N2—C8—C3 | −0.1 (2) | C9—N1—C17—C16 | −1.9 (3) |
| C6—C7—C8—N2 | −179.7 (2) | C10—N1—C17—C12 | 0.7 (2) |
| C6—C7—C8—C3 | −1.6 (3) | C9—N1—C17—C12 | 177.15 (18) |
| C4—C3—C8—N2 | −179.48 (17) | C15—C16—C17—N1 | 178.80 (18) |
| C2—C3—C8—N2 | 0.3 (2) | C15—C16—C17—C12 | −0.2 (3) |
| C4—C3—C8—C7 | 2.1 (3) | C13—C12—C17—N1 | −179.73 (16) |
| C2—C3—C8—C7 | −178.18 (18) | C11—C12—C17—N1 | −0.7 (2) |
| C10—N1—C9—C1 | −105.6 (2) | C13—C12—C17—C16 | −0.5 (3) |
| C17—N1—C9—C1 | 78.6 (3) | C11—C12—C17—C16 | 178.44 (17) |
| C2—C1—C9—N1 | −118.1 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| N2—H1*N*2···O1$^{i}$ | 0.88 (2) | 2.24 (3) | 3.037 (2) | 151 (2) |

Symmetry code: (i) $x-1$, $y$, $z$.