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# (*E*)-5,6-Dimethoxy-2-(pyridin-4-ylmethylidene)-2,3-dihydro-1*H*-inden-1-one

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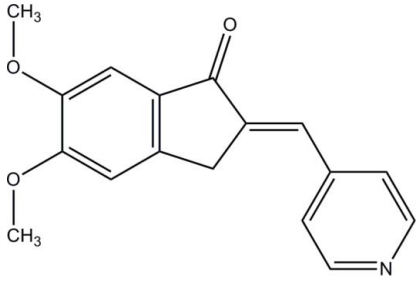
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.001 Å; $R$ factor = 0.039; $wR$ factor = 0.132; data-to-parameter ratio = 30.6.

The molecule of the title compound, $C_{17}H_{15}NO_3$, is slightly twisted, with a dihedral angle of 12.12 (3)° between the dihydroindenone group and the pyridine ring. In the crystal, molecules are connected into layers parallel to the *ab* plane *via* intermolecular C—H···O hydrogen bonds. Weak $\pi$–$\pi$ [centroid–centroid distance = 3.5680 (6) Å] interactions are also observed.

## Related literature

For general background and the biological activity of chalcone derivatives, see: Nowakowska (2008); Akihisa *et al.* (2006); Narender *et al.* (2005); Zhang *et al.* (2006); Dicarlo *et al.* (1999); Heidenreich *et al.* (2008); Syed *et al.* (2008); D'Archivio *et al.* (2008). For a related structure, see: Ali *et al.* (2010). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



## Experimental

### Crystal data

$C_{17}H_{15}NO_3$
$M_r$ = 281.30
Monoclinic, $P2_1/c$
$a$ = 10.7572 (14) Å
$b$ = 8.6057 (11) Å
$c$ = 17.2961 (17) Å
$\beta$ = 123.394 (6)°
$V$ = 1336.8 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 100 K
0.45 × 0.32 × 0.23 mm

### Data collection

Bruker APEXII Duo CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.958, $T_{max}$ = 0.979
21745 measured reflections
5874 independent reflections
5138 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.132
$S$ = 1.10
5874 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.61 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···$A$ |
|---|---|---|---|---|
| C14—H14$A$···O2$^{i}$ | 0.93 | 2.57 | 3.4787 (12) | 167 |
| C14—H14$A$···O3$^{i}$ | 0.93 | 2.57 | 3.2708 (12) | 133 |
| C16—H16$A$···O1$^{ii}$ | 0.96 | 2.53 | 3.0486 (11) | 114 |

Symmetry codes: (i) $x-1, y+1, z$; (ii) $-x+1, y-\frac{1}{2}, -z-\frac{1}{2}$.

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: RZ2492).

## References

Akihisa, T., Tokuda, H., Hasegawa, D., Ukiya, M., Kimura, Y. & Enjo, F. (2006). *J. Nat. Prod.* **69**, 38–42.
Ali, M. A., Ismail, R., Tan, S. C., Yeap, C. S. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o2531–o2532.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
D'Archivio, M., Santangelo, C., Scazzocchio, B., Vari, R., Filesi, C., Masella, R. & Giovannini, C. (2008). *Int. J. Mol. Sci.* **9**, 213–228.
Dicarlo, G., Mascolo, N., Izzo, A. A. & Capasso, F. (1999). *Life Sci.* **65**, 337–353.
Heidenreich, A., Aus, G., Bolla, M., Joniau, S., Matveev, V. B., Schmid, H. P. & Zattoni, F. (2008). *Eur. Urol.* **53**, 68–80.
Narender, T., Shweta, Tanvir, K., Srinivasa Rao, M., Srivastava, K. & Puri, S. K. (2005). *Bioorg. Med. Chem. Lett.* **15**, 2453–2455.
Nowakowska, Z. (2008). *Eur. J. Med. Chem.* **42**, 125–137.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Syed, D. N., Suh, Y., Afag, F. & Mukhtar, H. (2008). *Cancer Lett.* **265**, 167–176.
Zhang, Y., Shi, S., Zhao, M., Jiang, Y. & Tu, P. (2006). *Biochem. Syst. Ecol.* **34**, 766–769.

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§ Thomson Reuters ResearcherID: A-3561-2009.

# supporting information

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## (*E*)-5,6-Dimethoxy-2-(pyridin-4-ylmethylidene)-2,3-dihydro-1*H*-inden-1-one

**Mohamed Ashraf Ali, Rusli Ismail, Soo Choon Tan, Chin Sing Yeap and Hoong-Kun Fun**

### S1. Comment

Chalcone moieties are common substructures in numerous natural products belonging to the flavonoid family (Nowakowska, 2008; Akihisa *et al.* 2006; Narender *et al.*, 2005; Zhang *et al.*, 2006).Chalcone derivatives are very versatile as physiologically active compounds and substrates for the evaluation of various organic syntheses. Chalcones, one of the major classes of natural products with widespread distribution in spices, tea, beer, fruits and vegetables, have been recently subject of great interest for their pharmacological activities (Dicarlo *et al.*, 2009). Prostate cancer is one of the most commonly diagnosed cancers in men, and the second leading cause of cancer deaths in the European Union and United States of America (Heidenreich *et al.*, 2008). Many antitumor drugs have been developed for prostate cancer patients, but their intolerable systemic toxicity often limits their clinical use. Chemoprevention is one of the most promising approaches in prostate cancer research, in which natural or synthetic agents are used to prevent this malignant disease (Heidenreich *et al.*, 2008; Syed *et al.*, 2008; D′Archivio *et al.*, 2008).

The molecular structure of the title compound is slightly twisted (Fig. 1). The torsion angles of the two methoxy groups are [C16–O2–C4–C5] -175.13 (6) and [C17–O3–C5–C4] -178.48 (6)°. The maximum deviation of the dihydroindenone group is 0.028 (1) Å and it makes dihedral angle of 12.12 (3)° with the pyridine ring [C11–C13/N1/C14–C15]. The geometry parameters are comparable to those observed in a closely related structure (Ali *et al.*, 2010).

In the crystal structure, the molecules are linked together into chains by a bifurcated hydrogen bonds involving the intermolecular C14—H14A···O3 and C14—H14A···O3 hydrogen bonds (Table 1) generating a $R^2_1(5)$ ring motif. These chains are arranged in an anti-parallel layer (Fig. 2) and each pair of anti-parallel layers are interconnected into a two-dimensional plane parallel to *ab* plane *via* intermolecular C16—H16A···O1 hydrogen bonds (Fig. 3, Table 1). Weak $\pi\cdots\pi$ interactions are also observed [$Cg1\cdots Cg2^{iii}$ of 3.5680 (6) Å; (iii) 1 - *x*, 1 - *y*, -*z*. *Cg*1 and *Cg*2 are centroids of C1–C2/C7–C9 and C2–C7 ring, respectively].

### S2. Experimental

A mixture of 5,6-dimethoxy-2,3-dihydro-1*H*-indene-1-one (0.001 mmol) and isonicotinaldehyde (0.001 mmol) were dissolved in methanol (10 ml) and 30% sodium hydroxide solution (5 ml) was added and the solution stirred for 5 h. After completion of the reaction as evident from TLC, the mixture was poured into crushed ice then neutralized with concentrated HCl. The precipitated solid was filtered, washed with water and recrystallized from ethanol to reveal the title compound as light yellow crystals.

### S3. Refinement

All hydrogen atoms were positioned geometrically [C–H = 0.93–0.97 Å] and refined using a riding model with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for methyl H atoms. A rotating-group model was applied for the methyl groups.

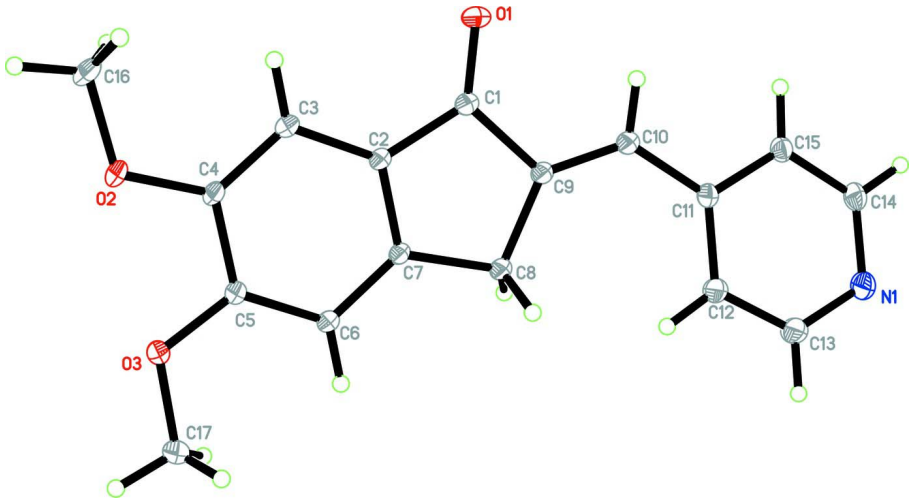


**Figure 1**

The molecular structure of the title compound with atom labels and 50% probability ellipsoids for non-H atoms.

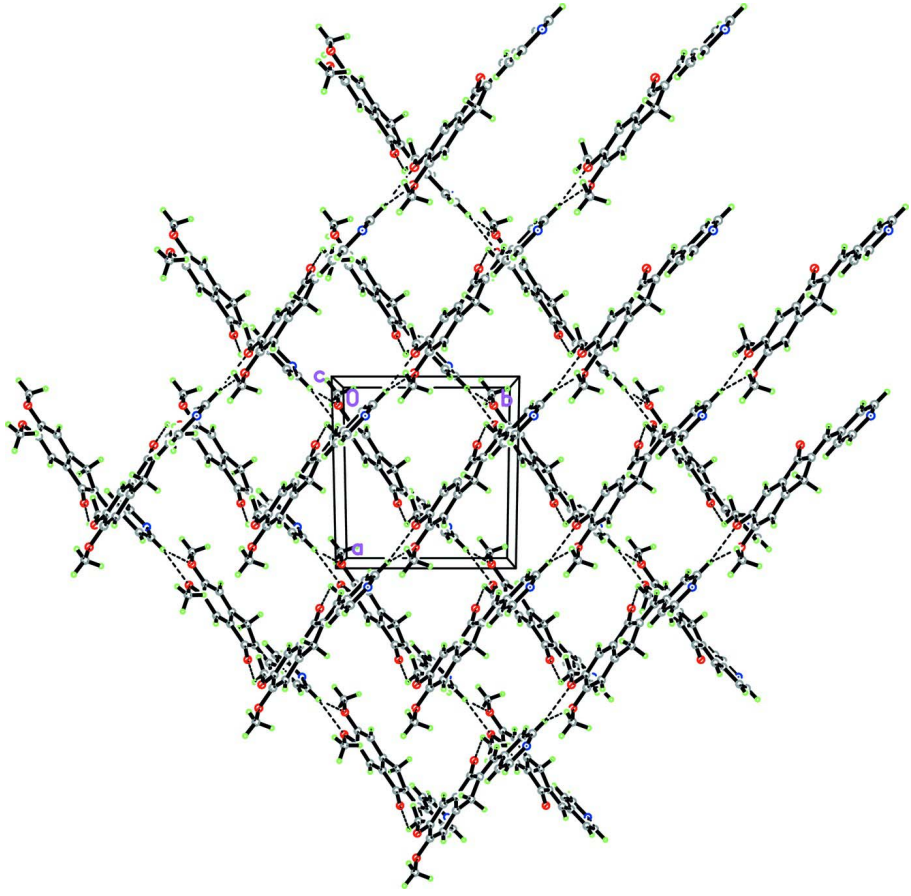


**Figure 2**

The crystal packing of title compound, showing chains arranged anti-parallelly. Intermolecular hydrogen bonds are shown as dashed lines.

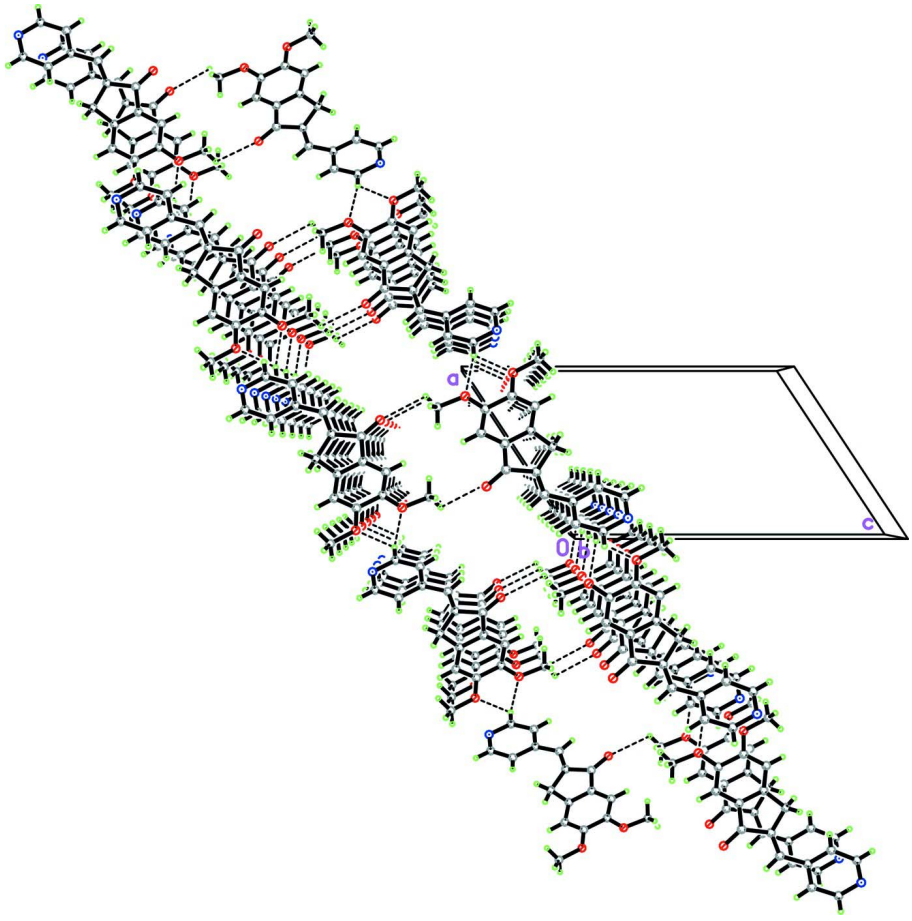

**Figure 3**

The crystal packing of title compound, showing a two-dimensional plane parallel to *ab* plane. Intermolecular hydrogen bonds are shown as dashed lines.

**(*E*)-5,6-Dimethoxy-2-(pyridin-4-ylmethylidene)- 2,3-dihydro-1*H*-inden-1-one**

*Crystal data*

$C_{17}H_{15}NO_3$
$M_r = 281.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.7572\ (14)$ Å
$b = 8.6057\ (11)$ Å
$c = 17.2961\ (17)$ Å
$\beta = 123.394\ (6)°$
$V = 1336.8\ (3)$ Å$^3$
$Z = 4$
$F(000) = 592$
$D_x = 1.398$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9933 reflections
$\theta = 2.8–35.1°$
$\mu = 0.10$ mm$^{-1}$
$T = 100$ K
Block, yellow
0.45 × 0.32 × 0.23 mm

*Data collection*

Bruker APEXII Duo CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.958$, $T_{max} = 0.979$
21745 measured reflections
5874 independent reflections

5138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 35.1°$, $\theta_{min} = 2.8°$
$h = -17 \rightarrow 17$
$k = -13 \rightarrow 13$
$l = -17 \rightarrow 27$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.132$
$S = 1.10$
5874 reflections
192 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2 + 0.2242P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.61$ 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 | 0.32457 (7) | 0.81081 (8) | −0.16256 (4) | 0.02006 (13) |
| O2 | 0.80183 (6) | 0.43182 (7) | −0.05682 (4) | 0.01665 (11) |
| O3 | 0.92680 (6) | 0.42839 (7) | 0.11848 (4) | 0.01546 (11) |
| N1 | 0.16589 (8) | 1.13656 (9) | 0.15678 (5) | 0.01914 (13) |
| C1 | 0.41258 (8) | 0.78448 (8) | −0.07968 (5) | 0.01273 (12) |
| C2 | 0.54736 (7) | 0.68903 (7) | −0.03693 (4) | 0.01082 (11) |
| C3 | 0.60522 (8) | 0.60592 (8) | −0.08026 (4) | 0.01211 (12) |
| H3A | 0.5587 | 0.6093 | −0.1442 | 0.015* |
| C4 | 0.73270 (7) | 0.51920 (8) | −0.02574 (4) | 0.01175 (12) |
| C5 | 0.80361 (7) | 0.51765 (8) | 0.07250 (4) | 0.01137 (11) |
| C6 | 0.74738 (7) | 0.60432 (8) | 0.11466 (4) | 0.01135 (11) |
| H6A | 0.7955 | 0.6055 | 0.1787 | 0.014* |
| C7 | 0.61696 (7) | 0.68941 (7) | 0.05839 (4) | 0.01039 (11) |
| C8 | 0.53304 (8) | 0.78599 (8) | 0.08831 (4) | 0.01196 (12) |
| H8A | 0.5018 | 0.7233 | 0.1213 | 0.014* |
| H8B | 0.5934 | 0.8717 | 0.1275 | 0.014* |
| C9 | 0.40070 (7) | 0.84391 (8) | −0.00227 (4) | 0.01164 (11) |
| C10 | 0.28495 (8) | 0.93423 (8) | −0.02118 (5) | 0.01312 (12) |
| H10A | 0.2176 | 0.9567 | −0.0835 | 0.016* |
| C11 | 0.24992 (7) | 1.00219 (8) | 0.04230 (5) | 0.01233 (12) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.31954 (9) | 0.96061 (10) | 0.13540 (5) | 0.01933 (15) |
| H12A | 0.3965 | 0.8883 | 0.1618 | 0.023* |
| C13 | 0.27285 (10) | 1.02821 (11) | 0.18809 (6) | 0.02310 (17) |
| H13A | 0.3188 | 0.9962 | 0.2493 | 0.028* |
| C14 | 0.10022 (8) | 1.17804 (9) | 0.06768 (5) | 0.01567 (13) |
| H14A | 0.0262 | 1.2536 | 0.0440 | 0.019* |
| C15 | 0.13705 (8) | 1.11388 (8) | 0.00888 (5) | 0.01385 (12) |
| H15A | 0.0866 | 1.1453 | −0.0527 | 0.017* |
| C16 | 0.74321 (9) | 0.43840 (10) | −0.15362 (5) | 0.01808 (14) |
| H16A | 0.8014 | 0.3730 | −0.1670 | 0.027* |
| H16B | 0.7471 | 0.5435 | −0.1708 | 0.027* |
| H16C | 0.6418 | 0.4032 | −0.1881 | 0.027* |
| C17 | 1.00081 (9) | 0.41893 (10) | 0.21693 (5) | 0.01939 (14) |
| H17A | 1.0849 | 0.3505 | 0.2415 | 0.029* |
| H17B | 0.9330 | 0.3794 | 0.2319 | 0.029* |
| H17C | 1.0342 | 0.5205 | 0.2434 | 0.029* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0204 (3) | 0.0275 (3) | 0.0098 (2) | 0.0087 (2) | 0.0067 (2) | 0.00324 (19) |
| O2 | 0.0182 (2) | 0.0220 (3) | 0.0120 (2) | 0.00551 (19) | 0.00975 (19) | −0.00182 (18) |
| O3 | 0.0142 (2) | 0.0200 (2) | 0.0111 (2) | 0.00625 (18) | 0.00627 (18) | 0.00077 (17) |
| N1 | 0.0189 (3) | 0.0226 (3) | 0.0175 (3) | 0.0056 (2) | 0.0109 (2) | −0.0015 (2) |
| C1 | 0.0138 (3) | 0.0142 (3) | 0.0106 (3) | 0.0021 (2) | 0.0070 (2) | 0.0008 (2) |
| C2 | 0.0119 (2) | 0.0118 (2) | 0.0096 (2) | 0.00066 (19) | 0.0065 (2) | −0.00052 (19) |
| C3 | 0.0132 (3) | 0.0140 (3) | 0.0095 (2) | 0.0003 (2) | 0.0065 (2) | −0.00105 (19) |
| C4 | 0.0130 (3) | 0.0135 (3) | 0.0106 (2) | 0.0005 (2) | 0.0076 (2) | −0.00177 (19) |
| C5 | 0.0115 (2) | 0.0128 (3) | 0.0104 (2) | 0.00140 (19) | 0.0064 (2) | −0.00031 (19) |
| C6 | 0.0122 (2) | 0.0129 (3) | 0.0095 (2) | 0.00100 (19) | 0.0063 (2) | −0.00042 (19) |
| C7 | 0.0118 (2) | 0.0111 (2) | 0.0094 (2) | 0.00076 (19) | 0.0066 (2) | −0.00013 (18) |
| C8 | 0.0136 (3) | 0.0131 (3) | 0.0101 (2) | 0.0024 (2) | 0.0071 (2) | 0.00030 (19) |
| C9 | 0.0131 (3) | 0.0122 (3) | 0.0105 (2) | 0.0015 (2) | 0.0071 (2) | 0.00038 (19) |
| C10 | 0.0138 (3) | 0.0144 (3) | 0.0115 (3) | 0.0024 (2) | 0.0072 (2) | 0.0007 (2) |
| C11 | 0.0121 (3) | 0.0128 (3) | 0.0129 (3) | 0.0015 (2) | 0.0074 (2) | −0.0003 (2) |
| C12 | 0.0212 (3) | 0.0241 (3) | 0.0141 (3) | 0.0106 (3) | 0.0105 (3) | 0.0025 (2) |
| C13 | 0.0249 (4) | 0.0309 (4) | 0.0149 (3) | 0.0132 (3) | 0.0118 (3) | 0.0027 (3) |
| C14 | 0.0146 (3) | 0.0147 (3) | 0.0185 (3) | 0.0023 (2) | 0.0096 (3) | −0.0008 (2) |
| C15 | 0.0131 (3) | 0.0139 (3) | 0.0154 (3) | 0.0019 (2) | 0.0083 (2) | 0.0009 (2) |
| C16 | 0.0212 (3) | 0.0235 (3) | 0.0127 (3) | 0.0006 (3) | 0.0113 (3) | −0.0035 (2) |
| C17 | 0.0184 (3) | 0.0259 (4) | 0.0118 (3) | 0.0083 (3) | 0.0070 (2) | 0.0035 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.2283 (8) | C8—H8A | 0.9700 |
| O2—C4 | 1.3593 (8) | C8—H8B | 0.9700 |
| O2—C16 | 1.4289 (9) | C9—C10 | 1.3465 (9) |
| O3—C5 | 1.3486 (8) | C10—C11 | 1.4646 (10) |

| | | | |
|---|---|---|---|
| O3—C17 | 1.4310 (9) | C10—H10A | 0.9300 |
| N1—C13 | 1.3419 (10) | C11—C12 | 1.3980 (10) |
| N1—C14 | 1.3427 (10) | C11—C15 | 1.3999 (10) |
| C1—C2 | 1.4640 (10) | C12—C13 | 1.3874 (11) |
| C1—C9 | 1.5029 (9) | C12—H12A | 0.9300 |
| C2—C7 | 1.3857 (9) | C13—H13A | 0.9300 |
| C2—C3 | 1.4050 (9) | C14—C15 | 1.3928 (10) |
| C3—C4 | 1.3796 (9) | C14—H14A | 0.9300 |
| C3—H3A | 0.9300 | C15—H15A | 0.9300 |
| C4—C5 | 1.4291 (9) | C16—H16A | 0.9600 |
| C5—C6 | 1.3926 (9) | C16—H16B | 0.9600 |
| C6—C7 | 1.3959 (9) | C16—H16C | 0.9600 |
| C6—H6A | 0.9300 | C17—H17A | 0.9600 |
| C7—C8 | 1.5124 (9) | C17—H17B | 0.9600 |
| C8—C9 | 1.5081 (9) | C17—H17C | 0.9600 |

| | | | |
|---|---|---|---|
| C4—O2—C16 | 117.18 (6) | C1—C9—C8 | 108.57 (5) |
| C5—O3—C17 | 117.17 (6) | C9—C10—C11 | 129.30 (6) |
| C13—N1—C14 | 116.14 (7) | C9—C10—H10A | 115.3 |
| O1—C1—C2 | 127.28 (6) | C11—C10—H10A | 115.3 |
| O1—C1—C9 | 126.02 (6) | C12—C11—C15 | 116.38 (6) |
| C2—C1—C9 | 106.69 (5) | C12—C11—C10 | 124.44 (6) |
| C7—C2—C3 | 121.87 (6) | C15—C11—C10 | 119.16 (6) |
| C7—C2—C1 | 109.63 (5) | C13—C12—C11 | 119.41 (7) |
| C3—C2—C1 | 128.51 (6) | C13—C12—H12A | 120.3 |
| C4—C3—C2 | 118.44 (6) | C11—C12—H12A | 120.3 |
| C4—C3—H3A | 120.8 | N1—C13—C12 | 124.49 (7) |
| C2—C3—H3A | 120.8 | N1—C13—H13A | 117.8 |
| O2—C4—C3 | 125.68 (6) | C12—C13—H13A | 117.8 |
| O2—C4—C5 | 114.52 (6) | N1—C14—C15 | 123.45 (7) |
| C3—C4—C5 | 119.80 (6) | N1—C14—H14A | 118.3 |
| O3—C5—C6 | 124.43 (6) | C15—C14—H14A | 118.3 |
| O3—C5—C4 | 114.50 (6) | C14—C15—C11 | 120.09 (7) |
| C6—C5—C4 | 121.07 (6) | C14—C15—H15A | 120.0 |
| C5—C6—C7 | 118.39 (6) | C11—C15—H15A | 120.0 |
| C5—C6—H6A | 120.8 | O2—C16—H16A | 109.5 |
| C7—C6—H6A | 120.8 | O2—C16—H16B | 109.5 |
| C2—C7—C6 | 120.38 (6) | H16A—C16—H16B | 109.5 |
| C2—C7—C8 | 112.02 (6) | O2—C16—H16C | 109.5 |
| C6—C7—C8 | 127.58 (6) | H16A—C16—H16C | 109.5 |
| C9—C8—C7 | 103.07 (5) | H16B—C16—H16C | 109.5 |
| C9—C8—H8A | 111.1 | O3—C17—H17A | 109.5 |
| C7—C8—H8A | 111.1 | O3—C17—H17B | 109.5 |
| C9—C8—H8B | 111.1 | H17A—C17—H17B | 109.5 |
| C7—C8—H8B | 111.1 | O3—C17—H17C | 109.5 |
| H8A—C8—H8B | 109.1 | H17A—C17—H17C | 109.5 |
| C10—C9—C1 | 120.04 (6) | H17B—C17—H17C | 109.5 |
| C10—C9—C8 | 131.39 (6) | | |

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|---|---|---|---|
| O1—C1—C2—C7 | 179.48 (7) | C5—C6—C7—C2 | −1.08 (10) |
| C9—C1—C2—C7 | −1.11 (8) | C5—C6—C7—C8 | 177.43 (6) |
| O1—C1—C2—C3 | −0.64 (12) | C2—C7—C8—C9 | 0.57 (7) |
| C9—C1—C2—C3 | 178.78 (7) | C6—C7—C8—C9 | −178.05 (7) |
| C7—C2—C3—C4 | 1.86 (10) | O1—C1—C9—C10 | 0.39 (12) |
| C1—C2—C3—C4 | −178.02 (7) | C2—C1—C9—C10 | −179.04 (6) |
| C16—O2—C4—C3 | 4.42 (10) | O1—C1—C9—C8 | −179.11 (7) |
| C16—O2—C4—C5 | −175.13 (6) | C2—C1—C9—C8 | 1.46 (7) |
| C2—C3—C4—O2 | 179.51 (6) | C7—C8—C9—C10 | 179.35 (7) |
| C2—C3—C4—C5 | −0.96 (10) | C7—C8—C9—C1 | −1.23 (7) |
| C17—O3—C5—C6 | 2.22 (10) | C1—C9—C10—C11 | 178.89 (7) |
| C17—O3—C5—C4 | −178.48 (6) | C8—C9—C10—C11 | −1.74 (13) |
| O2—C4—C5—O3 | −0.69 (9) | C9—C10—C11—C12 | −12.38 (13) |
| C3—C4—C5—O3 | 179.73 (6) | C9—C10—C11—C15 | 169.28 (7) |
| O2—C4—C5—C6 | 178.64 (6) | C15—C11—C12—C13 | 1.08 (12) |
| C3—C4—C5—C6 | −0.94 (10) | C10—C11—C12—C13 | −177.30 (8) |
| O3—C5—C6—C7 | −178.78 (6) | C14—N1—C13—C12 | 1.08 (14) |
| C4—C5—C6—C7 | 1.96 (10) | C11—C12—C13—N1 | −1.95 (15) |
| C3—C2—C7—C6 | −0.83 (10) | C13—N1—C14—C15 | 0.58 (12) |
| C1—C2—C7—C6 | 179.06 (6) | N1—C14—C15—C11 | −1.33 (12) |
| C3—C2—C7—C8 | −179.56 (6) | C12—C11—C15—C14 | 0.43 (11) |
| C1—C2—C7—C8 | 0.33 (8) | C10—C11—C15—C14 | 178.90 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14*A*···O2$^{i}$ | 0.93 | 2.57 | 3.4787 (12) | 167 |
| C14—H14*A*···O3$^{i}$ | 0.93 | 2.57 | 3.2708 (12) | 133 |
| C16—H16*A*···O1$^{ii}$ | 0.96 | 2.53 | 3.0486 (11) | 114 |

Symmetry codes: (i) *x*−1, *y*+1, *z*; (ii) −*x*+1, *y*−1/2, −*z*−1/2.