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# (5*R**)-5-[(2*S**,5*S**)-1-Methoxy-5-phenylpyrrolidin-2-yl]-3-methylfuran-2(5*H*)-one

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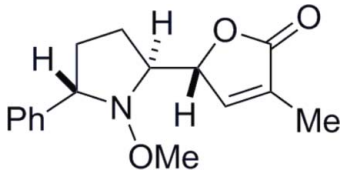
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.031; $wR$ factor = 0.075; data-to-parameter ratio = 8.2.

In the title compound, $C_{16}H_{19}NO_3$, the pyrrolidine ring is in a twist conformation. The dihedral angle between the dihydrofuran ring [maximum deviation = 0.0016 (11) Å] and the phenyl ring is 47.22 (8)°. In the crystal, molecules are linked by weak C—H···O hydrogen bonds, forming helical chains along the *b*-axis direction. The chains are further linked by C—H···$\pi$ interactions to constitute a three-dimensional architecture.

## Related literature

For noteworthy mild reactions of *N*-alkoxyamines, see: Hawker *et al.* (2001). For the reaction of Weinreb amide, see: Nahm & Weinreb (1981). For the synthesis of the title compound, see: Yoritate *et al.* (2014). For a related article utilizing similar compounds, see: Yanagita *et al.* (2013). For details of ring conformations, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{16}H_{19}NO_3$ $M_r$ = 273.32
Orthorhombic, $P2_12_12_1$
$a$ = 6.5427 (3) Å
$b$ = 10.8219 (5) Å
$c$ = 19.8397 (10) Å
$V$ = 1404.74 (12) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 90 K
0.54 × 0.51 × 0.40 mm

### Data collection

Bruker D8 diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min}$ = 0.95, $T_{max}$ = 0.97
12710 measured reflections
1510 independent reflections
1474 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.075
$S$ = 1.04
1510 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

*Cg*1 and *Cg*3 are the centroids of the O1/C2–C5 dihydrofuran and C15–C20 phenyl rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5···O6$^{i}$ | 1.00 | 2.51 | 3.185 (2) | 125 |
| C10—H10$A$···*Cg*1$^{ii}$ | 0.99 | 2.89 | 3.686 (2) | 138 |
| C16—H16···*Cg*3$^{iii}$ | 0.95 | 2.99 | 3.761 (2) | 139 |

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

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

We thank Professor S. Ohba (Keio University, Japan) for his valuable advice.

Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5367).

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# supporting information

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## (5*R**)-5-[(2*S**,5*S**)-1-Methoxy-5-phenylpyrrolidin-2-yl]-3-methylfuran-2(5*H*)-one

**Takeshi Oishi, Makoto Yoritate, Takaaki Sato and Noritaka Chida**

### S1. Comment

A number of compounds containing oxidized nitrogen functionality have been widely used in organic synthesis. In these substances, the *N*-alkoxyamines are known as the initiators for the stable free radical polymerization (Hawker *et al.*, 2001), and the *N*-alkoxyamides are utilized for mild and effective acylating agents (*cf.* Weinreb amide; Nahm & Weinreb, 1981). We noticed this inert N—O covalent bond, to develop a novel reaction to synthesize the natural alkaloids (Yanagita *et al.*, 2013).

In the title compound, the dihydrofuran ring is planar with a maximum deviation of 0.0016 (11) Å at atom C4, and the pyrrolidine ring is in a twist conformation with puckering parameters of Q(2) = 0.4145 (18) Å and $\varphi(2)$ = 10.6 (3)° (Cremer & Pople, 1975). Atoms N8 and C9 are deviated by –0.4566 (13) and 0.1991 (19) Å, respectively, from the plane of other carbon atoms (C10–C12). Angles of O13—N8—C9, O13—N8—C12 and C9—N8—C12 being 110.28 (13), 108.44 (12) and 106.87 (13)°, respectively, revealed the $sp^3$ configuration of the N8 atom. The relative configurations were confirmed by the X-ray analysis as C5*R*, C9*S* and C12*S*.

The crystal packing iss stabilized by an intermolecular C5—H5···O6 (–*x* + 1, *y* + 1/2, –*z* + 3/2) hydrogen bond (Table 1), forming a helical chain along to the [010] direction (Fig. 2). Further intermolecular C—H···$\pi$ interactions form a three-dimensional network in the crystal structure (Fig. 3). Distances for C10—H10A···*Cg*1 (*x* – 1, *y*, *z*) and C16—H···*Cg*3 (*x* + 1/2, –*y* + 1/2, –*z* + 1) are 3.686 (2) and 3.761 (2) Å, respectively. *Cg*1 and *Cg*3 are the centroids of the O1/C2–C5 dihydrofuran and C15–C20 phenyl rings, respectively. Additionally, weak intramolecular interactions, C12—H···O1, C5—H···O13 and C10—H10B···*Cg*1 being 2.957 (2), 2.791 (2) and 2.963 (2) Å, respectively, adopt the molecule into a sterically hindered conformation. The C5—O1 bond of dihydrofuran is overhanged on the pyrrolidine ring, with torsion angles of O1—C5—C9—N8 and O1—C5—C9—C10 being –69.7 (2) and 46.5 (2)°, respectively (Fig. 4).

### S2. Experimental

The title compound was synthesized from 4-oxo-4-phenylbutyric acid (Yoritate *et al.*, 2014), and recrystallized from a toluene solution by slow evaporation at ambient temperature; M.p. 358.5–359.9 K (not corrected). $^1$H NMR (500 MHz, $CDCl_3$) $\delta$ (p.p.m.) = 7.41–7.37 (m, 2H, Ph), 7.36–7.31 (m, 2H, Ph), 7.29–7.24 (m, 1H, Ph), 7.13 (qd, *J* = 1.7, 1.7 Hz, 1H, H4), 5.35–5.31 (m, 1H, H5), 4.33 (dd, *J* = 8.2, 7.5 Hz, 1H, H12), 3.56 (ddd, *J* = 8.3, 4.9, 4.9 Hz, 1H, H9), 3.35 (s, 3H, OMe), 2.20 (dddd, *J* = 12.9, 10.0, 7.5, 4.0 Hz, 1H, H11A), 2.00 (dddd, *J* = 13.1, 10.3, 8.3, 4.0 Hz, 1H, H10A), 1.95 (dd, *J* = 1.7, 1.7 Hz, 3H, CMe), 1.93–1.84 (m, 1H, H11B), 1.62 (dddd, *J* = 13.1, 10.0, 6.6, 4.9 Hz, 1H, H10B); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ (p.p.m.) = 174.6 (C), 148.1 (CH), 141.1 (C), 130.7 (C), 128.3 (CH), 128.1 (CH), 127.4 (CH), 80.5 (CH), 68.6 (CH), 65.3 (CH), 61.2 ($CH_2$), 28.9 ($CH_2$), 22.6 ($CH_2$), 10.9 ($CH_3$); Anal. calcd. for $C_{16}H_{19}NO_3$: C 70.31, H 7.01, N 5.12%, found: C 70.15, H 7.00, N 5.06%.

### S3. Refinement

C-bound H atoms were positioned geometrically with C—H = 0.95–1.00 Å, and constrained to ride on their parent atoms with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C). The Friedel opposites were merged before the final refinement because no significant anomalous dispersion was observed and the Flack parameter was a meaningless value of –1.2 (10) with 1054 Bijvoet pairs. One reflection (7 3 4) has been omitted in the final refinement.

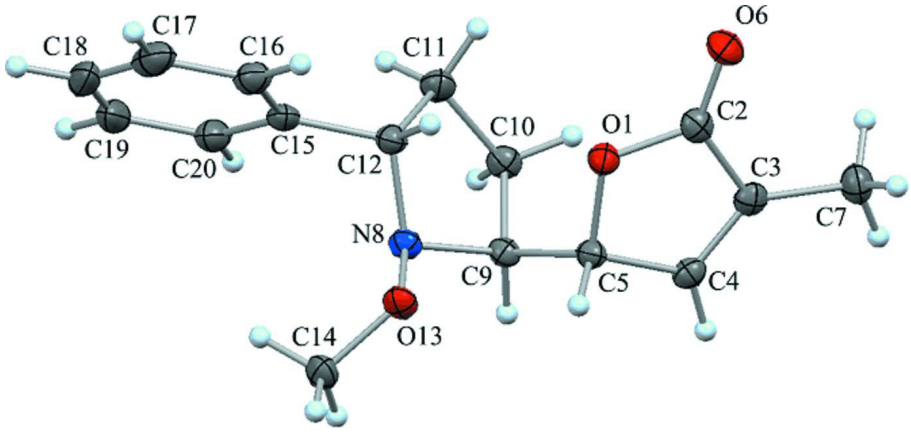


**Figure 1**

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

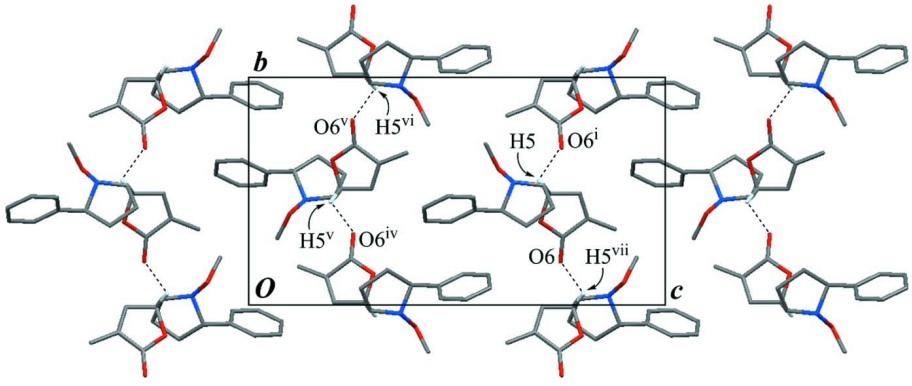


**Figure 2**

The crystal packing of the title compound, viewed down the *a* axis. Dashed lines indicate the intermolecular C5—H···O6 interactions, making helical chains along [010]. Only H atoms involved in hydrogen bonds were shown for clarity. Symmetry codes: (i) –*x* + 1, *y* + 1/2, –*z* + 3/2; (iv) *x* – 1/2, –*y* + 1/2, –*z* + 1; (v) –*x* + 1/2, –*y* + 1, *z* – 1/2; (vi) *x* – 1/2, –*y* + 3/2, –*z* + 1; (vii) –*x* + 1, *y* – 1/2, –*z* + 3/2.

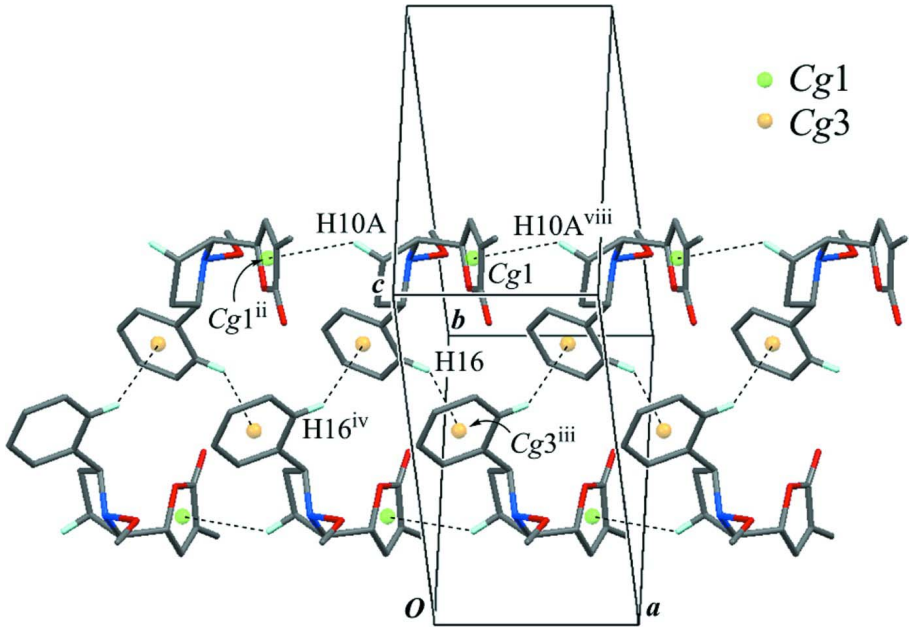


**Figure 3**

A view for the intermolecular C—H···$\pi$ interactions (dashed lines), showing parallel (C10—H10A···*Cg*1) and alternated (C16—H16···*Cg*3) chains along [100]. *Cg*1 and *Cg*3 are the centroids of the O1/C2–C5 dihydrofuran and the C15–C20 phenyl rings, respectively. Only H atoms involved in hydrogen bonds were shown for clarity. Symmetry codes: (ii) $x - 1$, $y$, $z$; (iii) $x + 1/2$, $-y + 1/2$, $-z + 1$; (iv) $x - 1/2$, $-y + 1/2$, $-z + 1$; (viii) $x + 1$, $y$, $z$.

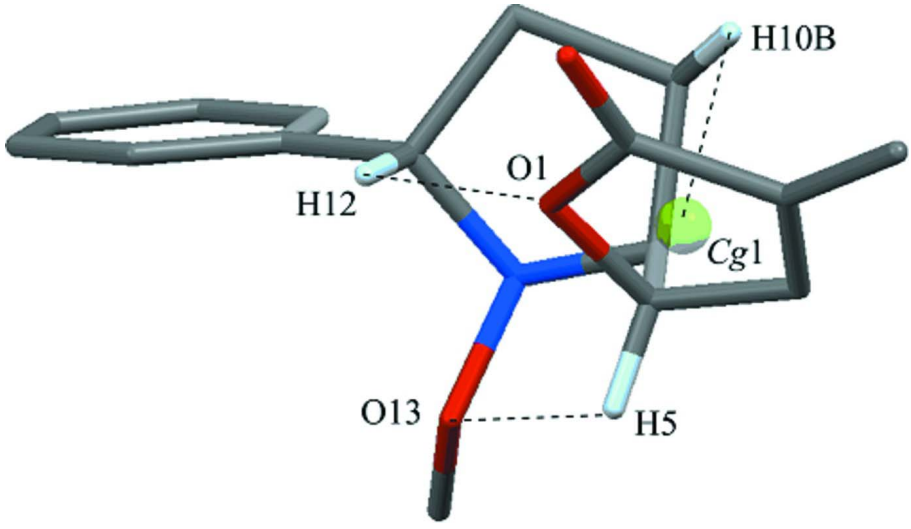


**Figure 4**

Molecular conformation indicating intramolecular C—H···O and C—H···$\pi$ interactions with dashed lines. *Cg*1 is a centroid of the O1/C2–C5 dihydrofuran ring.

**(5*R**)-5-[(2*S**,5*S**)-1-Methoxy-5-phenylpyrrolidin-2-yl]-3-methylfuran-2(5*H*)-one**

*Crystal data*

$C_{16}H_{19}NO_3$
$M_r = 273.32$
Orthorhombic, $P2_12_12_1$
$a = 6.5427$ (3) Å
$b = 10.8219$ (5) Å
$c = 19.8397$ (10) Å
$V = 1404.74$ (12) Å$^3$
$Z = 4$
$F(000) = 584$
$D_x = 1.292$ Mg m$^{-3}$
Melting point: 358.5 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9968 reflections
$\theta = 2.8$–$25.4°$
$\mu = 0.09$ mm$^{-1}$
$T = 90$ K
Prism, colourless
0.54 × 0.51 × 0.40 mm

*Data collection*

Bruker D8 diffractometer
Radiation source: fine-focus sealed tube
Multilayered confocal mirror monochromator
Detector resolution: 8.333 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min} = 0.95$, $T_{max} = 0.97$
12710 measured reflections
1510 independent reflections
1474 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.8°$
$h = -7 \rightarrow 7$
$k = -13 \rightarrow 11$
$l = -23 \rightarrow 22$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.075$
$S = 1.04$
1510 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.402P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.014$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ e Å$^{-3}$
Extinction correction: *SHELXL*
Extinction coefficient: 0.029 (3)

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.28934 (19) | 0.36404 (11) | 0.70066 (5) | 0.0197 (3) |
| C2 | 0.3603 (3) | 0.29777 (16) | 0.75407 (8) | 0.0200 (4) |
| C3 | 0.3704 (3) | 0.37890 (16) | 0.81354 (8) | 0.0198 (4) |

| | | | | |
|---|---|---|---|---|
| C4 | 0.3059 (3) | 0.48928 (16) | 0.79475 (8) | 0.0198 (4) |
| H4 | 0.2965 | 0.559 | 0.8237 | 0.024* |
| C5 | 0.2496 (3) | 0.48950 (17) | 0.72169 (8) | 0.0181 (4) |
| H5 | 0.3424 | 0.5469 | 0.6967 | 0.022* |
| O6 | 0.4060 (2) | 0.19033 (12) | 0.74867 (6) | 0.0268 (3) |
| C7 | 0.4522 (3) | 0.33239 (18) | 0.87893 (8) | 0.0272 (4) |
| H7A | 0.4346 | 0.3959 | 0.9137 | 0.041* |
| H7B | 0.5978 | 0.3132 | 0.874 | 0.041* |
| H7C | 0.3779 | 0.2576 | 0.8921 | 0.041* |
| N8 | −0.0284 (2) | 0.53897 (13) | 0.63598 (7) | 0.0185 (3) |
| C9 | 0.0267 (3) | 0.52517 (16) | 0.70776 (8) | 0.0179 (4) |
| H9 | −0.0038 | 0.6045 | 0.7316 | 0.021* |
| C10 | −0.1287 (3) | 0.42769 (16) | 0.73027 (8) | 0.0209 (4) |
| H10A | −0.2569 | 0.4672 | 0.7457 | 0.025* |
| H10B | −0.0728 | 0.3768 | 0.7674 | 0.025* |
| C11 | −0.1671 (3) | 0.34798 (17) | 0.66697 (8) | 0.0245 (4) |
| H11A | −0.1119 | 0.2636 | 0.6735 | 0.029* |
| H11B | −0.3154 | 0.3418 | 0.6576 | 0.029* |
| C12 | −0.0570 (3) | 0.41297 (15) | 0.60873 (8) | 0.0193 (4) |
| H12 | 0.0794 | 0.3736 | 0.6014 | 0.023* |
| O13 | 0.13547 (19) | 0.59737 (11) | 0.59960 (6) | 0.0208 (3) |
| C14 | 0.0574 (3) | 0.70653 (17) | 0.56898 (9) | 0.0251 (4) |
| H14A | −0.0573 | 0.6851 | 0.5394 | 0.038* |
| H14B | 0.1653 | 0.7461 | 0.5424 | 0.038* |
| H14C | 0.0103 | 0.7636 | 0.604 | 0.038* |
| C15 | −0.1725 (3) | 0.41689 (16) | 0.54286 (8) | 0.0205 (4) |
| C16 | −0.0888 (3) | 0.36521 (18) | 0.48516 (8) | 0.0258 (4) |
| H16 | 0.0411 | 0.3262 | 0.4873 | 0.031* |
| C17 | −0.1934 (4) | 0.36997 (19) | 0.42435 (9) | 0.0358 (5) |
| H17 | −0.1347 | 0.3345 | 0.385 | 0.043* |
| C18 | −0.3824 (4) | 0.42609 (19) | 0.42089 (10) | 0.0398 (6) |
| H18 | −0.4529 | 0.4303 | 0.3791 | 0.048* |
| C19 | −0.4695 (4) | 0.47615 (19) | 0.47816 (11) | 0.0370 (5) |
| H19 | −0.6006 | 0.5136 | 0.4758 | 0.044* |
| C20 | −0.3655 (3) | 0.47190 (18) | 0.53920 (10) | 0.0276 (4) |
| H20 | −0.4257 | 0.5064 | 0.5785 | 0.033* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0201 (6) | 0.0197 (6) | 0.0194 (5) | 0.0030 (5) | −0.0008 (5) | −0.0022 (5) |
| C2 | 0.0136 (8) | 0.0222 (9) | 0.0241 (8) | 0.0007 (7) | 0.0023 (7) | 0.0019 (7) |
| C3 | 0.0147 (8) | 0.0230 (8) | 0.0218 (8) | −0.0029 (8) | 0.0017 (7) | 0.0009 (7) |
| C4 | 0.0176 (9) | 0.0211 (8) | 0.0208 (8) | −0.0024 (8) | 0.0002 (7) | −0.0018 (7) |
| C5 | 0.0195 (9) | 0.0159 (8) | 0.0188 (8) | −0.0004 (7) | 0.0000 (7) | 0.0002 (6) |
| O6 | 0.0274 (7) | 0.0209 (6) | 0.0321 (6) | 0.0070 (6) | −0.0013 (6) | −0.0006 (5) |
| C7 | 0.0262 (10) | 0.0310 (10) | 0.0243 (8) | 0.0009 (9) | −0.0032 (8) | 0.0054 (8) |
| N8 | 0.0179 (7) | 0.0181 (7) | 0.0194 (7) | −0.0025 (6) | 0.0022 (6) | 0.0024 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0182 (9) | 0.0168 (8) | 0.0187 (8) | 0.0017 (7) | 0.0014 (7) | 0.0000 (7) |
| C10 | 0.0175 (8) | 0.0214 (9) | 0.0237 (8) | −0.0005 (8) | 0.0027 (7) | 0.0025 (7) |
| C11 | 0.0253 (10) | 0.0231 (9) | 0.0251 (8) | −0.0063 (8) | −0.0019 (8) | 0.0039 (7) |
| C12 | 0.0198 (9) | 0.0159 (8) | 0.0223 (8) | 0.0000 (7) | −0.0003 (7) | 0.0002 (7) |
| O13 | 0.0178 (6) | 0.0212 (6) | 0.0235 (6) | −0.0030 (5) | 0.0019 (5) | 0.0052 (5) |
| C14 | 0.0280 (10) | 0.0247 (9) | 0.0227 (8) | −0.0052 (8) | −0.0044 (8) | 0.0078 (7) |
| C15 | 0.0238 (9) | 0.0151 (8) | 0.0226 (8) | −0.0051 (8) | −0.0028 (7) | 0.0015 (6) |
| C16 | 0.0297 (10) | 0.0222 (9) | 0.0254 (8) | −0.0071 (9) | 0.0004 (8) | 0.0003 (7) |
| C17 | 0.0543 (14) | 0.0300 (10) | 0.0231 (8) | −0.0180 (12) | −0.0020 (9) | 0.0010 (8) |
| C18 | 0.0557 (15) | 0.0305 (11) | 0.0330 (10) | −0.0178 (11) | −0.0238 (11) | 0.0103 (9) |
| C19 | 0.0349 (12) | 0.0220 (10) | 0.0541 (13) | −0.0045 (9) | −0.0221 (11) | 0.0069 (9) |
| C20 | 0.0268 (10) | 0.0198 (9) | 0.0362 (10) | −0.0017 (9) | −0.0062 (9) | −0.0002 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.361 (2) | C11—C12 | 1.532 (2) |
| O1—C5 | 1.444 (2) | C11—H11A | 0.99 |
| C2—O6 | 1.205 (2) | C11—H11B | 0.99 |
| C2—C3 | 1.472 (2) | C12—C15 | 1.510 (2) |
| C3—C4 | 1.321 (3) | C12—H12 | 1.0 |
| C3—C7 | 1.491 (2) | O13—C14 | 1.423 (2) |
| C4—C5 | 1.495 (2) | C14—H14A | 0.98 |
| C4—H4 | 0.95 | C14—H14B | 0.98 |
| C5—C9 | 1.534 (3) | C14—H14C | 0.98 |
| C5—H5 | 1.0 | C15—C16 | 1.387 (2) |
| C7—H7A | 0.98 | C15—C20 | 1.398 (3) |
| C7—H7B | 0.98 | C16—C17 | 1.388 (3) |
| C7—H7C | 0.98 | C16—H16 | 0.95 |
| N8—O13 | 1.4385 (19) | C17—C18 | 1.380 (4) |
| N8—C9 | 1.477 (2) | C17—H17 | 0.95 |
| N8—C12 | 1.479 (2) | C18—C19 | 1.382 (3) |
| C9—C10 | 1.532 (2) | C18—H18 | 0.95 |
| C9—H9 | 1.0 | C19—C20 | 1.390 (3) |
| C10—C11 | 1.544 (2) | C19—H19 | 0.95 |
| C10—H10A | 0.99 | C20—H20 | 0.95 |
| C10—H10B | 0.99 | | |
| | | | |
| C2—O1—C5 | 109.38 (12) | C12—C11—C10 | 106.28 (14) |
| O6—C2—O1 | 121.57 (16) | C12—C11—H11A | 110.5 |
| O6—C2—C3 | 129.46 (17) | C10—C11—H11A | 110.5 |
| O1—C2—C3 | 108.97 (14) | C12—C11—H11B | 110.5 |
| C4—C3—C2 | 107.40 (14) | C10—C11—H11B | 110.5 |
| C4—C3—C7 | 131.73 (16) | H11A—C11—H11B | 108.7 |
| C2—C3—C7 | 120.80 (16) | N8—C12—C15 | 110.73 (13) |
| C3—C4—C5 | 110.71 (15) | N8—C12—C11 | 101.97 (13) |
| C3—C4—H4 | 124.6 | C15—C12—C11 | 115.48 (15) |
| C5—C4—H4 | 124.6 | N8—C12—H12 | 109.5 |
| O1—C5—C4 | 103.55 (14) | C15—C12—H12 | 109.5 |

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| O1—C5—C9 | 110.83 (14) | C11—C12—H12 | 109.5 |
| C4—C5—C9 | 114.14 (15) | C14—O13—N8 | 108.14 (13) |
| O1—C5—H5 | 109.4 | O13—C14—H14A | 109.5 |
| C4—C5—H5 | 109.4 | O13—C14—H14B | 109.5 |
| C9—C5—H5 | 109.4 | H14A—C14—H14B | 109.5 |
| C3—C7—H7A | 109.5 | O13—C14—H14C | 109.5 |
| C3—C7—H7B | 109.5 | H14A—C14—H14C | 109.5 |
| H7A—C7—H7B | 109.5 | H14B—C14—H14C | 109.5 |
| C3—C7—H7C | 109.5 | C16—C15—C20 | 119.04 (17) |
| H7A—C7—H7C | 109.5 | C16—C15—C12 | 120.38 (17) |
| H7B—C7—H7C | 109.5 | C20—C15—C12 | 120.58 (16) |
| O13—N8—C9 | 110.28 (13) | C15—C16—C17 | 120.55 (19) |
| O13—N8—C12 | 108.44 (12) | C15—C16—H16 | 119.7 |
| C9—N8—C12 | 106.87 (13) | C17—C16—H16 | 119.7 |
| N8—C9—C10 | 100.89 (14) | C18—C17—C16 | 120.1 (2) |
| N8—C9—C5 | 115.55 (14) | C18—C17—H17 | 120.0 |
| C10—C9—C5 | 113.92 (14) | C16—C17—H17 | 120.0 |
| N8—C9—H9 | 108.7 | C17—C18—C19 | 120.08 (19) |
| C10—C9—H9 | 108.7 | C17—C18—H18 | 120.0 |
| C5—C9—H9 | 108.7 | C19—C18—H18 | 120.0 |
| C9—C10—C11 | 104.81 (13) | C18—C19—C20 | 120.1 (2) |
| C9—C10—H10A | 110.8 | C18—C19—H19 | 119.9 |
| C11—C10—H10A | 110.8 | C20—C19—H19 | 119.9 |
| C9—C10—H10B | 110.8 | C19—C20—C15 | 120.10 (19) |
| C11—C10—H10B | 110.8 | C19—C20—H20 | 120.0 |
| H10A—C10—H10B | 108.9 | C15—C20—H20 | 120.0 |

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| C5—O1—C2—O6 | −179.35 (17) | C9—C10—C11—C12 | −7.74 (19) |
| C5—O1—C2—C3 | −0.06 (19) | O13—N8—C12—C15 | −77.91 (17) |
| O6—C2—C3—C4 | 179.44 (19) | C9—N8—C12—C15 | 163.22 (14) |
| O1—C2—C3—C4 | 0.2 (2) | O13—N8—C12—C11 | 158.69 (13) |
| O6—C2—C3—C7 | 2.1 (3) | C9—N8—C12—C11 | 39.81 (18) |
| O1—C2—C3—C7 | −177.16 (15) | C10—C11—C12—N8 | −18.38 (18) |
| C2—C3—C4—C5 | −0.3 (2) | C10—C11—C12—C15 | −138.50 (15) |
| C7—C3—C4—C5 | 176.70 (18) | C9—N8—O13—C14 | −122.23 (14) |
| C2—O1—C5—C4 | −0.11 (18) | C12—N8—O13—C14 | 121.07 (14) |
| C2—O1—C5—C9 | −122.92 (14) | N8—C12—C15—C16 | 123.43 (18) |
| C3—C4—C5—O1 | 0.25 (19) | C11—C12—C15—C16 | −121.35 (18) |
| C3—C4—C5—C9 | 120.85 (17) | N8—C12—C15—C20 | −57.0 (2) |
| O13—N8—C9—C10 | −162.41 (13) | C11—C12—C15—C20 | 58.3 (2) |
| C12—N8—C9—C10 | −44.74 (17) | C20—C15—C16—C17 | 1.2 (3) |
| O13—N8—C9—C5 | −39.07 (19) | C12—C15—C16—C17 | −179.20 (17) |
| C12—N8—C9—C5 | 78.60 (18) | C15—C16—C17—C18 | −0.2 (3) |
| O1—C5—C9—N8 | −69.67 (17) | C16—C17—C18—C19 | −0.9 (3) |
| C4—C5—C9—N8 | 173.89 (15) | C17—C18—C19—C20 | 1.0 (3) |
| O1—C5—C9—C10 | 46.50 (18) | C18—C19—C20—C15 | 0.0 (3) |
| C4—C5—C9—C10 | −69.9 (2) | C16—C15—C20—C19 | −1.1 (3) |
| N8—C9—C10—C11 | 30.75 (17) | C12—C15—C20—C19 | 179.31 (17) |

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| C5—C9—C10—C11 | −93.70 (17) |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 and *Cg*3 are the centroids of the O1/C2–C5 dihydrofuran and C15–C20 phenyl rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C12—H12···O1 | 1.00 | 2.40 | 2.957 (2) | 114 |
| C5—H5···O13 | 1.00 | 2.42 | 2.791 (2) | 101 |
| C10—H10*B*···*Cg*1 | 0.99 | 2.56 | 2.963 (2) | 104 |
| C5—H5···O6$^{i}$ | 1.00 | 2.51 | 3.185 (2) | 125 |
| C10—H10*A*···*Cg*1$^{ii}$ | 0.99 | 2.89 | 3.686 (2) | 138 |
| C16—H16···*Cg*3$^{iii}$ | 0.95 | 2.99 | 3.761 (2) | 139 |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*; (iii) *x*+1/2, −*y*+1/2, −*z*+1.