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# 3-Isopropyl-2,6-bis(4-methoxyphenyl)-piperidin-4-one

**K. Ravichandran,[a] S. Sethuvasan,[b] K. Thirunavukarasu,[b] S. Ponnuswamy[b]* and M. N. Ponnuswamy[a]**

[a]Centre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and [b]Department of Chemistry, Government Arts College (Autonomous), Coimbatore 641 018, India
Correspondence e-mail: mnpsy2004@yahoo.com

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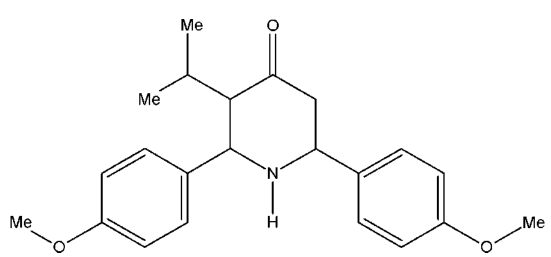
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.044; $wR$ factor = 0.121; data-to-parameter ratio = 11.6.

In the title compound, $C_{22}H_{27}NO_3$, the piperidine ring adopts a slightly distorted chair conformation. The dihedral angle between the two aromatic rings is 60.4 (1)°. In the crystal, the amino group forms a rather long N—H⋯O contact to a methoxy O atom. There are also C—H⋯O interactions present.

## Related literature

For the biological activity of piperidine derivatives, see: Bochringer & Soehne (1961); El-Subbagh *et al.* (2000); Ganellin & Spickett (1965); Hagenbach & Gysin (1952); Jerom & Spencer (1988); Katritzky & Fan (1990); Perumal *et al.* (2001); Ravindran *et al.* (1991); Severs *et al.* (1965). For puckering parameters, see: Cremer & Pople (1975). For asymmetry parameters, see: Nardelli (1983). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).




## Experimental

*Crystal data*

$C_{22}H_{27}NO_3$
$M_r$ = 353.45
Orthorhombic, $P2_12_12_1$
$a$ = 7.5547 (3) Å
$b$ = 11.8792 (6) Å
$c$ = 22.1103 (10) Å
$V$ = 1984.26 (16) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 K
0.22 × 0.20 × 0.18 mm

*Data collection*

Bruker SMART APEX CCD detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008) $T_{min}$ = 0.983, $T_{max}$ = 0.986
10956 measured reflections
2801 independent reflections
1835 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.121
$S$ = 1.03
2801 reflections
241 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H1⋯O1$^{i}$ | 0.90 (2) | 2.66 (2) | 3.538 (2) | 167.4 (17) |
| C16—H16$A$⋯O1$^{ii}$ | 0.96 | 2.57 | 3.474 (4) | 156 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5928).

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# supporting information

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## 3-Isopropyl-2,6-bis(4-methoxyphenyl)piperidin-4-one

**K. Ravichandran, S. Sethuvasan, K. Thirunavukarasu, S. Ponnuswamy and M. N. Ponnuswamy**

### S1. Comment

In the family of heterocyclic compounds, piperidin-4-ones possess varied biological properties such as antiviral, antitumour (El-Subbagh *et al.*, 2000), analgesic (Jerom & Spencer, 1988), local anaesthetic (Perumal *et al.*, 2001; Hagenbach & Gysin, 1952), anti-inflammatory and anticancer activities (Katritzky & Fan, 1990). Several 2,6-disubstituted piperidines are found to be useful as tranquillisers (Bochringer & Soehne, 1961) and possess hypotensive activity (Severs *et al.*, 1965), a combination of stimulant and depressant effects on the central nervous system (Ganellin & Spickett, 1965). Also the substitution of methoxy phenyl groups at 2,6-positions is found to be active against *CNS* subpanels. In addition, the bulkiness of the subtituent in different positons of the piperidine ring leads to the decrease in carcinogenecity (Ravindran *et al.*, 1991). In view of the importance, the crystallographic study of the title compound has been carried out to establish the molecular structure and conformation.

The *ORTEP* plot of the molecule is shown in Fig. 1. The piperidine ring adopts distorted chair conformation. The puckering (Cremer & Pople, 1975) and the asymmetry parameters (Nardelli, 1983) are: $q_2$=0.121 (3) Å, $q_3$ = 0.569 (3) Å, $\varphi_2$ = 7.6 (1)° and $\Delta_s$(N1 & C4) = 1.3 (2)°. The sum of the bond angles around the atom N1 [333.5°] is in accordance with $sp^3$ hybridization.

The best plane of the piperidine ring is oriented with respect to the phenyl rings (C9—C14) & (C18—C23) at angles of 82.8 (1)° & 86.2 (1)°, respectively. The two phenyl rings are set apart with an angle of 60.4 (1)°. The methoxy groups substituted at the phenyl rings are coplanar, which can be seen from the torsion angles of [C13—C12—O1—C7=] 4.4 (4)° for (C9—C14) ring and [C20—C21—O3—C8 =] 1.6 (5)° for (C18—C23) ring.

The packing of the molecules is stabilized by a rather long N-H···O contact and by C—H···O interactions in addition to van der Waals forces.

### S2. Experimental

Ammonium acetate (100 mmol), anisaldehyde (200 mmol) and isobutylmethylketone (100 mmol) in ethanol (30 ml) were heated on a hot plate at 50–55° C and after the completion of reaction, water was added and extracted with ether, dried and recrystallized from ethanol.

### S3. Refinement

Due to the absence of anomalous scatterers, the absolute configuration could not be determined and Friedel pairs were merged. C-bound H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms and 1.2$U_{eq}$(C) for all other H atoms. The H atom bonded to N was freely refined.

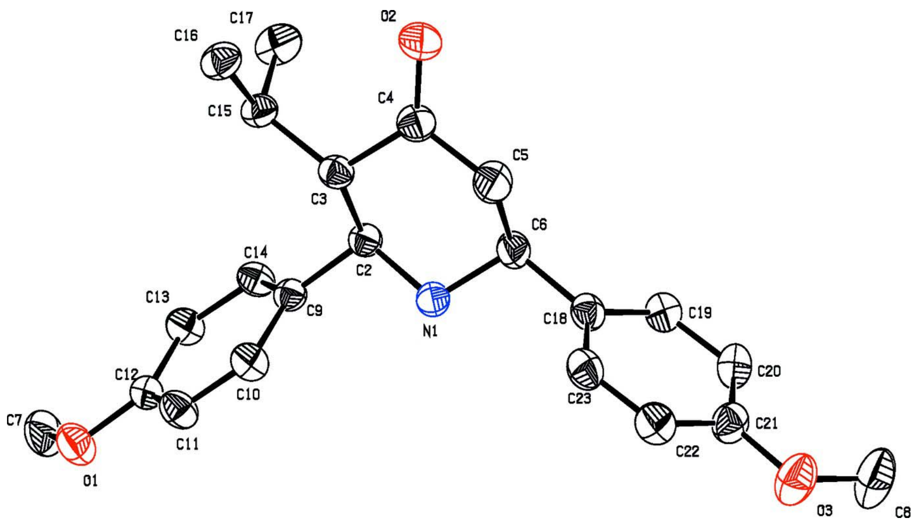


**Figure 1**

The molecular structure of the title compound, showing the atomic numbering and displacement ellipsoids drawn at 30% probability level.

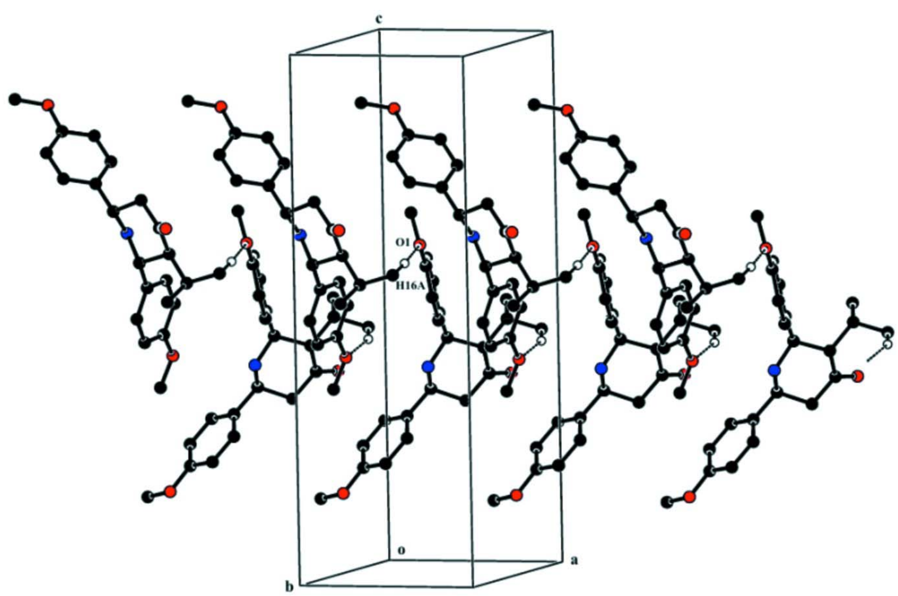


**Figure 2**

The crystal packing of the molecules viewed down *b* axis. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

## 3-Isopropyl-2,6-bis(4-methoxyphenyl)piperidin-4-one

### *Crystal data*

$C_{22}H_{27}NO_3$
$M_r$ = 353.45
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 7.5547 (3) Å
$b$ = 11.8792 (6) Å
$c$ = 22.1103 (10) Å
$V$ = 1984.26 (16) Å$^3$
$Z$ = 4
$F$(000) = 760
$D_x$ = 1.183 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å

Cell parameters from 2938 reflections
$\theta = 1.8–28.3°$
$\mu = 0.08\ mm^{-1}$
$T = 293$ K
Black, white crystalline
0.22 × 0.20 × 0.18 mm

*Data collection*

Bruker SMART APEX CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.983$, $T_{max} = 0.986$
10956 measured reflections
2801 independent reflections
1835 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.8°$
$h = -10 \rightarrow 9$
$k = -15 \rightarrow 8$
$l = -27 \rightarrow 29$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.121$
$S = 1.03$
2801 reflections
241 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
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.0599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.015$
$\Delta\rho_{max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{min} = -0.13$ e Å$^{-3}$

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| H1 | 0.161 (4) | 0.568 (2) | 0.1225 (11) | 0.049 (7)* |
| O1 | 0.3703 (3) | 0.80940 (17) | −0.10981 (8) | 0.0655 (6) |
| O2 | 0.6143 (3) | 0.2871 (2) | 0.15552 (10) | 0.0773 (7) |
| O3 | −0.2117 (3) | 0.5919 (2) | 0.37078 (10) | 0.0865 (8) |
| N1 | 0.2620 (3) | 0.53392 (18) | 0.13369 (9) | 0.0446 (5) |
| C2 | 0.3533 (3) | 0.4891 (2) | 0.08036 (10) | 0.0433 (6) |
| H2 | 0.2910 | 0.4212 | 0.0669 | 0.052* |
| C3 | 0.5444 (3) | 0.4554 (2) | 0.09959 (11) | 0.0451 (6) |
| H3 | 0.5993 | 0.5241 | 0.1155 | 0.054* |
| C4 | 0.5323 (4) | 0.3749 (3) | 0.15235 (13) | 0.0556 (7) |
| C5 | 0.4118 (4) | 0.4096 (3) | 0.20247 (12) | 0.0676 (9) |
| H5A | 0.4656 | 0.4711 | 0.2247 | 0.081* |

| | | | | |
|---|---|---|---|---|
| H5B | 0.3966 | 0.3470 | 0.2302 | 0.081* |
| C6 | 0.2300 (3) | 0.4469 (2) | 0.17908 (12) | 0.0507 (6) |
| H6 | 0.1737 | 0.3825 | 0.1592 | 0.061* |
| C7 | 0.3092 (5) | 0.7822 (3) | −0.16890 (12) | 0.0731 (9) |
| H7A | 0.3785 | 0.7215 | −0.1850 | 0.110* |
| H7B | 0.3206 | 0.8469 | −0.1946 | 0.110* |
| H7C | 0.1872 | 0.7599 | −0.1670 | 0.110* |
| C8 | −0.3714 (6) | 0.5342 (4) | 0.38404 (18) | 0.1054 (14) |
| H8A | −0.4477 | 0.5363 | 0.3493 | 0.158* |
| H8B | −0.4292 | 0.5699 | 0.4176 | 0.158* |
| H8C | −0.3453 | 0.4574 | 0.3941 | 0.158* |
| C9 | 0.3520 (3) | 0.5733 (2) | 0.02940 (10) | 0.0413 (5) |
| C10 | 0.4059 (3) | 0.6843 (2) | 0.03831 (12) | 0.0508 (6) |
| H10 | 0.4390 | 0.7076 | 0.0768 | 0.061* |
| C11 | 0.4112 (4) | 0.7599 (2) | −0.00879 (13) | 0.0555 (7) |
| H11 | 0.4486 | 0.8333 | −0.0019 | 0.067* |
| C12 | 0.3614 (3) | 0.7275 (2) | −0.06612 (11) | 0.0470 (6) |
| C13 | 0.3080 (4) | 0.6181 (2) | −0.07623 (11) | 0.0518 (7) |
| H13 | 0.2749 | 0.5950 | −0.1148 | 0.062* |
| C14 | 0.3041 (3) | 0.5433 (2) | −0.02846 (11) | 0.0493 (6) |
| H14 | 0.2676 | 0.4697 | −0.0357 | 0.059* |
| C15 | 0.6625 (3) | 0.4165 (2) | 0.04695 (12) | 0.0470 (6) |
| H15 | 0.6556 | 0.4757 | 0.0162 | 0.056* |
| C16 | 0.8571 (3) | 0.4099 (3) | 0.06599 (13) | 0.0578 (7) |
| H16A | 0.8910 | 0.4792 | 0.0851 | 0.087* |
| H16B | 0.9296 | 0.3976 | 0.0309 | 0.087* |
| H16C | 0.8728 | 0.3488 | 0.0939 | 0.087* |
| C17 | 0.6047 (4) | 0.3087 (3) | 0.01612 (14) | 0.0691 (8) |
| H17A | 0.4828 | 0.3150 | 0.0043 | 0.104* |
| H17B | 0.6181 | 0.2467 | 0.0436 | 0.104* |
| H17C | 0.6765 | 0.2961 | −0.0191 | 0.104* |
| C18 | 0.1107 (3) | 0.4866 (2) | 0.22900 (11) | 0.0489 (6) |
| C19 | −0.0469 (3) | 0.4320 (2) | 0.24044 (12) | 0.0521 (6) |
| H19 | −0.0796 | 0.3720 | 0.2159 | 0.063* |
| C20 | −0.1579 (4) | 0.4634 (3) | 0.28710 (12) | 0.0577 (7) |
| H20 | −0.2627 | 0.4243 | 0.2939 | 0.069* |
| C21 | −0.1124 (4) | 0.5531 (3) | 0.32367 (12) | 0.0579 (7) |
| C22 | 0.0451 (4) | 0.6089 (3) | 0.31305 (13) | 0.0625 (8) |
| H22 | 0.0774 | 0.6689 | 0.3377 | 0.075* |
| C23 | 0.1547 (4) | 0.5767 (2) | 0.26643 (13) | 0.0577 (7) |
| H23 | 0.2597 | 0.6156 | 0.2598 | 0.069* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0953 (15) | 0.0511 (11) | 0.0502 (11) | −0.0043 (11) | 0.0027 (10) | 0.0050 (9) |
| O2 | 0.0696 (13) | 0.0697 (14) | 0.0927 (15) | 0.0253 (12) | 0.0052 (11) | 0.0272 (12) |
| O3 | 0.0879 (16) | 0.1049 (19) | 0.0665 (14) | −0.0023 (15) | 0.0205 (12) | −0.0176 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0394 (10) | 0.0469 (13) | 0.0475 (12) | 0.0030 (10) | −0.0020 (9) | 0.0051 (10) |
| C2 | 0.0364 (10) | 0.0433 (14) | 0.0503 (13) | −0.0006 (10) | −0.0025 (10) | 0.0002 (11) |
| C3 | 0.0401 (11) | 0.0420 (14) | 0.0532 (14) | 0.0022 (10) | −0.0056 (11) | 0.0021 (13) |
| C4 | 0.0427 (13) | 0.0603 (18) | 0.0638 (17) | 0.0085 (13) | −0.0080 (12) | 0.0113 (15) |
| C5 | 0.0638 (17) | 0.085 (2) | 0.0543 (16) | 0.0188 (17) | −0.0014 (14) | 0.0201 (16) |
| C6 | 0.0504 (13) | 0.0529 (16) | 0.0487 (14) | 0.0018 (12) | 0.0016 (11) | 0.0082 (13) |
| C7 | 0.108 (2) | 0.068 (2) | 0.0435 (16) | 0.0003 (19) | 0.0086 (17) | 0.0070 (15) |
| C8 | 0.096 (3) | 0.137 (4) | 0.083 (2) | −0.012 (3) | 0.038 (2) | −0.014 (2) |
| C9 | 0.0333 (10) | 0.0407 (14) | 0.0499 (14) | 0.0004 (10) | −0.0019 (10) | −0.0008 (11) |
| C10 | 0.0588 (15) | 0.0479 (16) | 0.0457 (14) | −0.0043 (13) | −0.0080 (12) | −0.0018 (13) |
| C11 | 0.0693 (17) | 0.0400 (15) | 0.0572 (16) | −0.0103 (13) | −0.0047 (14) | −0.0029 (13) |
| C12 | 0.0519 (13) | 0.0414 (16) | 0.0478 (14) | 0.0024 (12) | 0.0057 (11) | 0.0012 (12) |
| C13 | 0.0661 (16) | 0.0472 (16) | 0.0420 (14) | 0.0008 (12) | −0.0071 (12) | −0.0036 (12) |
| C14 | 0.0567 (14) | 0.0383 (14) | 0.0529 (15) | −0.0021 (11) | −0.0056 (12) | −0.0028 (12) |
| C15 | 0.0413 (11) | 0.0454 (14) | 0.0543 (14) | 0.0048 (11) | −0.0024 (11) | −0.0031 (12) |
| C16 | 0.0431 (13) | 0.0596 (18) | 0.0706 (18) | 0.0027 (12) | −0.0004 (13) | −0.0089 (15) |
| C17 | 0.0547 (16) | 0.0677 (19) | 0.085 (2) | 0.0008 (15) | −0.0062 (15) | −0.0205 (17) |
| C18 | 0.0478 (13) | 0.0529 (16) | 0.0461 (13) | 0.0025 (12) | −0.0039 (11) | 0.0104 (13) |
| C19 | 0.0509 (13) | 0.0556 (16) | 0.0498 (14) | −0.0027 (13) | −0.0045 (12) | 0.0008 (13) |
| C20 | 0.0513 (13) | 0.073 (2) | 0.0485 (14) | −0.0068 (14) | 0.0014 (12) | 0.0042 (14) |
| C21 | 0.0625 (15) | 0.0669 (19) | 0.0443 (14) | 0.0046 (14) | 0.0016 (13) | 0.0002 (14) |
| C22 | 0.0728 (18) | 0.0581 (19) | 0.0566 (17) | −0.0064 (15) | −0.0047 (15) | −0.0039 (15) |
| C23 | 0.0561 (15) | 0.0556 (17) | 0.0615 (17) | −0.0095 (14) | −0.0029 (13) | 0.0068 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C12 | 1.373 (3) | C10—C11 | 1.376 (4) |
| O1—C7 | 1.423 (3) | C10—H10 | 0.9300 |
| O2—C4 | 1.215 (3) | C11—C12 | 1.377 (4) |
| O3—C21 | 1.364 (3) | C11—H11 | 0.9300 |
| O3—C8 | 1.419 (5) | C12—C13 | 1.379 (4) |
| N1—C6 | 1.461 (3) | C13—C14 | 1.381 (4) |
| N1—C2 | 1.466 (3) | C13—H13 | 0.9300 |
| N1—H1 | 0.90 (3) | C14—H14 | 0.9300 |
| C2—C9 | 1.507 (3) | C15—C17 | 1.515 (4) |
| C2—C3 | 1.557 (3) | C15—C16 | 1.531 (3) |
| C2—H2 | 0.9800 | C15—H15 | 0.9800 |
| C3—C4 | 1.512 (4) | C16—H16A | 0.9600 |
| C3—C15 | 1.538 (3) | C16—H16B | 0.9600 |
| C3—H3 | 0.9800 | C16—H16C | 0.9600 |
| C4—C5 | 1.493 (4) | C17—H17A | 0.9600 |
| C5—C6 | 1.533 (4) | C17—H17B | 0.9600 |
| C5—H5A | 0.9700 | C17—H17C | 0.9600 |
| C5—H5B | 0.9700 | C18—C19 | 1.380 (4) |
| C6—C18 | 1.501 (4) | C18—C23 | 1.392 (4) |
| C6—H6 | 0.9800 | C19—C20 | 1.381 (4) |
| C7—H7A | 0.9600 | C19—H19 | 0.9300 |
| C7—H7B | 0.9600 | C20—C21 | 1.382 (4) |

| | | | |
|---|---|---|---|
| C7—H7C | 0.9600 | C20—H20 | 0.9300 |
| C8—H8A | 0.9600 | C21—C22 | 1.382 (4) |
| C8—H8B | 0.9600 | C22—C23 | 1.377 (4) |
| C8—H8C | 0.9600 | C22—H22 | 0.9300 |
| C9—C14 | 1.377 (3) | C23—H23 | 0.9300 |
| C9—C10 | 1.393 (4) | | |

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| C12—O1—C7 | 118.0 (2) | C9—C10—H10 | 119.4 |
| C21—O3—C8 | 117.5 (3) | C10—C11—C12 | 120.4 (3) |
| C6—N1—C2 | 111.9 (2) | C10—C11—H11 | 119.8 |
| C6—N1—H1 | 111.7 (16) | C12—C11—H11 | 119.8 |
| C2—N1—H1 | 109.9 (16) | O1—C12—C11 | 115.9 (2) |
| N1—C2—C9 | 110.95 (19) | O1—C12—C13 | 124.6 (2) |
| N1—C2—C3 | 108.06 (19) | C11—C12—C13 | 119.5 (2) |
| C9—C2—C3 | 112.40 (18) | C12—C13—C14 | 119.3 (2) |
| N1—C2—H2 | 108.4 | C12—C13—H13 | 120.4 |
| C9—C2—H2 | 108.4 | C14—C13—H13 | 120.4 |
| C3—C2—H2 | 108.4 | C9—C14—C13 | 122.6 (2) |
| C4—C3—C15 | 115.4 (2) | C9—C14—H14 | 118.7 |
| C4—C3—C2 | 108.5 (2) | C13—C14—H14 | 118.7 |
| C15—C3—C2 | 114.1 (2) | C17—C15—C16 | 111.0 (2) |
| C4—C3—H3 | 106.0 | C17—C15—C3 | 115.3 (2) |
| C15—C3—H3 | 106.0 | C16—C15—C3 | 111.4 (2) |
| C2—C3—H3 | 106.0 | C17—C15—H15 | 106.2 |
| O2—C4—C5 | 120.4 (3) | C16—C15—H15 | 106.2 |
| O2—C4—C3 | 123.9 (3) | C3—C15—H15 | 106.2 |
| C5—C4—C3 | 115.8 (2) | C15—C16—H16A | 109.5 |
| C4—C5—C6 | 112.1 (2) | C15—C16—H16B | 109.5 |
| C4—C5—H5A | 109.2 | H16A—C16—H16B | 109.5 |
| C6—C5—H5A | 109.2 | C15—C16—H16C | 109.5 |
| C4—C5—H5B | 109.2 | H16A—C16—H16C | 109.5 |
| C6—C5—H5B | 109.2 | H16B—C16—H16C | 109.5 |
| H5A—C5—H5B | 107.9 | C15—C17—H17A | 109.5 |
| N1—C6—C18 | 112.5 (2) | C15—C17—H17B | 109.5 |
| N1—C6—C5 | 106.8 (2) | H17A—C17—H17B | 109.5 |
| C18—C6—C5 | 112.4 (2) | C15—C17—H17C | 109.5 |
| N1—C6—H6 | 108.4 | H17A—C17—H17C | 109.5 |
| C18—C6—H6 | 108.4 | H17B—C17—H17C | 109.5 |
| C5—C6—H6 | 108.4 | C19—C18—C23 | 117.3 (2) |
| O1—C7—H7A | 109.5 | C19—C18—C6 | 120.4 (2) |
| O1—C7—H7B | 109.5 | C23—C18—C6 | 122.4 (2) |
| H7A—C7—H7B | 109.5 | C18—C19—C20 | 122.3 (3) |
| O1—C7—H7C | 109.5 | C18—C19—H19 | 118.9 |
| H7A—C7—H7C | 109.5 | C20—C19—H19 | 118.9 |
| H7B—C7—H7C | 109.5 | C19—C20—C21 | 119.6 (3) |
| O3—C8—H8A | 109.5 | C19—C20—H20 | 120.2 |
| O3—C8—H8B | 109.5 | C21—C20—H20 | 120.2 |
| H8A—C8—H8B | 109.5 | O3—C21—C22 | 116.2 (3) |

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| O3—C8—H8C | 109.5 | O3—C21—C20 | 124.8 (3) |
| H8A—C8—H8C | 109.5 | C22—C21—C20 | 119.0 (3) |
| H8B—C8—H8C | 109.5 | C23—C22—C21 | 120.8 (3) |
| C14—C9—C10 | 117.0 (2) | C23—C22—H22 | 119.6 |
| C14—C9—C2 | 121.6 (2) | C21—C22—H22 | 119.6 |
| C10—C9—C2 | 121.3 (2) | C22—C23—C18 | 121.0 (3) |
| C11—C10—C9 | 121.3 (2) | C22—C23—H23 | 119.5 |
| C11—C10—H10 | 119.4 | C18—C23—H23 | 119.5 |

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| C6—N1—C2—C9 | 168.49 (19) | C10—C11—C12—C13 | −0.7 (4) |
| C6—N1—C2—C3 | −67.9 (2) | O1—C12—C13—C14 | −179.8 (2) |
| N1—C2—C3—C4 | 54.9 (3) | C11—C12—C13—C14 | 0.5 (4) |
| C9—C2—C3—C4 | 177.7 (2) | C10—C9—C14—C13 | −0.1 (4) |
| N1—C2—C3—C15 | −174.9 (2) | C2—C9—C14—C13 | −177.6 (2) |
| C9—C2—C3—C15 | −52.1 (3) | C12—C13—C14—C9 | −0.1 (4) |
| C15—C3—C4—O2 | 3.0 (4) | C4—C3—C15—C17 | 61.6 (3) |
| C2—C3—C4—O2 | 132.5 (3) | C2—C3—C15—C17 | −65.1 (3) |
| C15—C3—C4—C5 | −177.4 (2) | C4—C3—C15—C16 | −66.0 (3) |
| C2—C3—C4—C5 | −47.9 (3) | C2—C3—C15—C16 | 167.3 (2) |
| O2—C4—C5—C6 | −131.9 (3) | N1—C6—C18—C19 | 120.8 (3) |
| C3—C4—C5—C6 | 48.5 (4) | C5—C6—C18—C19 | −118.7 (3) |
| C2—N1—C6—C18 | −170.4 (2) | N1—C6—C18—C23 | −61.0 (3) |
| C2—N1—C6—C5 | 65.9 (3) | C5—C6—C18—C23 | 59.5 (3) |
| C4—C5—C6—N1 | −53.8 (3) | C23—C18—C19—C20 | −0.5 (4) |
| C4—C5—C6—C18 | −177.6 (3) | C6—C18—C19—C20 | 177.9 (2) |
| N1—C2—C9—C14 | −131.8 (2) | C18—C19—C20—C21 | 0.7 (4) |
| C3—C2—C9—C14 | 107.1 (3) | C8—O3—C21—C22 | −178.0 (3) |
| N1—C2—C9—C10 | 50.7 (3) | C8—O3—C21—C20 | 1.6 (5) |
| C3—C2—C9—C10 | −70.4 (3) | C19—C20—C21—O3 | 179.7 (3) |
| C14—C9—C10—C11 | −0.2 (4) | C19—C20—C21—C22 | −0.8 (4) |
| C2—C9—C10—C11 | 177.4 (2) | O3—C21—C22—C23 | −179.7 (3) |
| C9—C10—C11—C12 | 0.6 (4) | C20—C21—C22—C23 | 0.7 (4) |
| C7—O1—C12—C11 | −175.9 (3) | C21—C22—C23—C18 | −0.5 (4) |
| C7—O1—C12—C13 | 4.4 (4) | C19—C18—C23—C22 | 0.4 (4) |
| C10—C11—C12—O1 | 179.6 (2) | C6—C18—C23—C22 | −177.9 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.90 (2) | 2.66 (2) | 3.538 (2) | 167.4 (17) |
| C16—H16*A*···O1$^{ii}$ | 0.96 | 2.57 | 3.474 (4) | 156 |

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