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

# 4,4,5,5-Tetramethyl-2-(3,4,5-trimethoxyphenyl)imidazolidine-1-oxyl 3-oxide

**Hai-Bo Wang, Lin-Lin Jing, Peng Gao and Xiao-Li Sun***

Department of Chemistry, School of Pharmacy, Fourth Military Medical University, Changle West Road 17, 710032 Xi-An, People's Republic of China
Correspondence e-mail: lisydcn@yahoo.com.cn

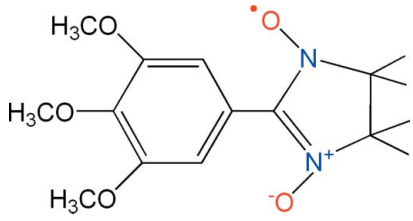
Received 26 June 2009; accepted 23 July 2009

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.046; $wR$ factor = 0.119; data-to-parameter ratio = 14.0.

In the title nitronyl nitroxide radical compound, $C_{16}C_{23}N_2O_5$, the imidazole and benzene rings are twisted with respect to each other, making a dihedral angle of 26.2 (4)°. The imidazole ring adopts a half-chair conformation. Weak C—H$\cdots\pi$ interactions are also found.

## Related literature

For the preparation of the title compound see: Ullman *et al.* (1974). For related structures, see: Feher *et al.* (2008); Gao *et al.* (2009); Qin *et al.* (2009); Cirujeda *et al.* (1995); Matsushita *et al.* (1997). For the coordination properties of the title compound and its use in the formation of molecule-based magnetic materials, see: Takui *et al.* (2009). For puckering parameters, see: Cremer & Pople (1975).



## Experimental

### Crystal data

$C_{16}H_{23}N_2O_5$
$M_r$ = 323.36
Orthorhombic, *Pbca*
$a$ = 20.623 (3) Å
$b$ = 7.2168 (12) Å
$c$ = 22.831 (4) Å
$V$ = 3398.0 (10) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.32 × 0.25 × 0.17 mm

### Data collection

Bruker APEXII diffractometer
Absorption correction: none
15858 measured reflections
3027 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.062

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.119
$S$ = 1.06
3027 reflections
216 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C14—H14$A\cdots Cg2^{i}$ | 0.96 | 2.80 | 3.644 (2) | 147 |

Symmetry code: (i) $-x, -y, -z+1$. $Cg2$ is the centroid of the phenyl ring.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

We thank the Natural Science Foundation of China (grant No. 20802092) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2472).

## References

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Cirujeda, J., Mas, M., Molins, E., Panthou de, F. L., Laugier, J., Park, J. G., Paulsen, C., Rey, P., Rovira, C. & Veciana, J. (1995). *Chem. Commun*. pp. 709–710.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc*. **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Feher, R., Wurst, K., Amabilino, D. B. & Veciana, J. (2008). *Inorg. Chim. Acta*, **361**, 4094–4099.
Gao, Z.-Y., Chang, J.-L., Xian, D. & Jiang, K. (2009). *Acta Cryst.* E**65**, o1062.
Matsushita, M. M., Izuoka, A., Sugawara, T., Kobayashi, T., Wada, N., Takeda, N. & Ishikawa, M. (1997). *J. Am. Chem. Soc*. **119**, 4369–4379.
Qin, X.-Y., Wang, P.-A. & Sun, X.-L. (2009). *Acta Cryst.* E**65**, o1031.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Takui, T., Hosokoshi, Y., Lan, X., Miyazaki, Y., Inada, A. & Okada, K. (2009). *J. Am. Chem. Soc*. **131**, 4670–4673.
Ullman, E. F., Osiecki, J. H., Boocock, D. G. B. & Darcy, R. (1974). *J. Am. Chem. Soc*. **96**, 7049–7053.

# supporting information

*Acta Cryst.* (2009). E**65**, o2090 [doi:10.1107/S1600536809029274]

## 4,4,5,5-Tetramethyl-2-(3,4,5-trimethoxyphenyl)imidazolidine-1-oxyl 3-oxide

**Hai-Bo Wang, Lin-Lin Jing, Peng Gao and Xiao-Li Sun**

### S1. Comment

The title radical compound was obtained from the oxidation of 4,4,5,5- tetramethyl-2-(3,4,5-trimethoxybenzenyl)-imidazolidine-1,3-diol, which was prepared by the condensation of 3,4,5-trimethoxybenzaldehyde with 2,3-Dimethyl-2,3-bis(hydroxyl-amino)butane. The title compound was used for coordination with many metal cations, such as $Mn^{2+}$, $Cu^{2+}$, and $Ni^{2+}$, in order to form some molecule-based magnetic materials (Takui *et al.*, 2009).

The molecular structure of the title compound is shown in Fig 1. Examination of bond length within the five membered rings shows an average structure as observed with related compounds (Cirujeda *et al.*, 1995; Feher *et al.*, 2008; Gao *et al.*, 2009; Matsushita *et al.*, 1997; Qin *et al.*, 2009).

The imidazole and the phenyl rings are twisted with respect to each other making a dihedral angle of 26.2 (4)°. The imidazole ring has an half-chair conformation with puckering parameters O(2)=. 0.0275 (2)Å and $\varphi$= 233.0 (5)°(Cremer & Pople, 1975). The crystal structure is stabilized by weak C—H$\cdots\pi$ (Table 1, *Cg*2 is the centroid of the phenyl ring) and van der Waals interactions.

### S2. Experimental

The compound 4,4,5,5-tetramethyl-2-(3,4,5-trimethoxybenzenyl)-imidazolidine -1-oxyl-3-oxide was prepared according to the method reported by Ullman *et al.* (1974). 2,3-Dimethyl-2,3-bis(hydroxylamino) butane (1.48 g, 10.0 mmol) and 3,4,5-trimethoxybenzaldehyde (1.96 g, 10.0 mmol) were dissolved in a methanol-water mixture (2:1), which was stirred for 5 h at reflux temperature, then cooled to room temperature and filtered. The white powder was washed by methanol. This product was dried under vaccum, then, it was suspended in dichloromethane (50.0 ml) and the water solution (30.0 ml) of $NaIO_4$ (1.7 g) was added and stirred at ice bath for 20 min. The reaction mixture was extracted by dichloromethane (30.0 ml) for twice and the organic layer was combined and dried over $Na_2SO_4$. Then the solvent was removed to give a dark blue residue which was purified by a flash column chromatography (eluent, ether and petroleum ether, the ratio of volume is 2 to 1) to yield the title compound (I) as a dark blue powder. Single crystals of compound (I) were obtained from the mixed solution of *n*-heptane and dichloromethane (the ratio of volume is 1 to 1).

### S3. Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl) or 0.93 Å (aromatic) with $U_{iso}$(H) = 1.2$U_{eq}$($C_{aromatic}$) or $U_{iso}$(H) = 1.5$U_{eq}$($C_{methyl}$).

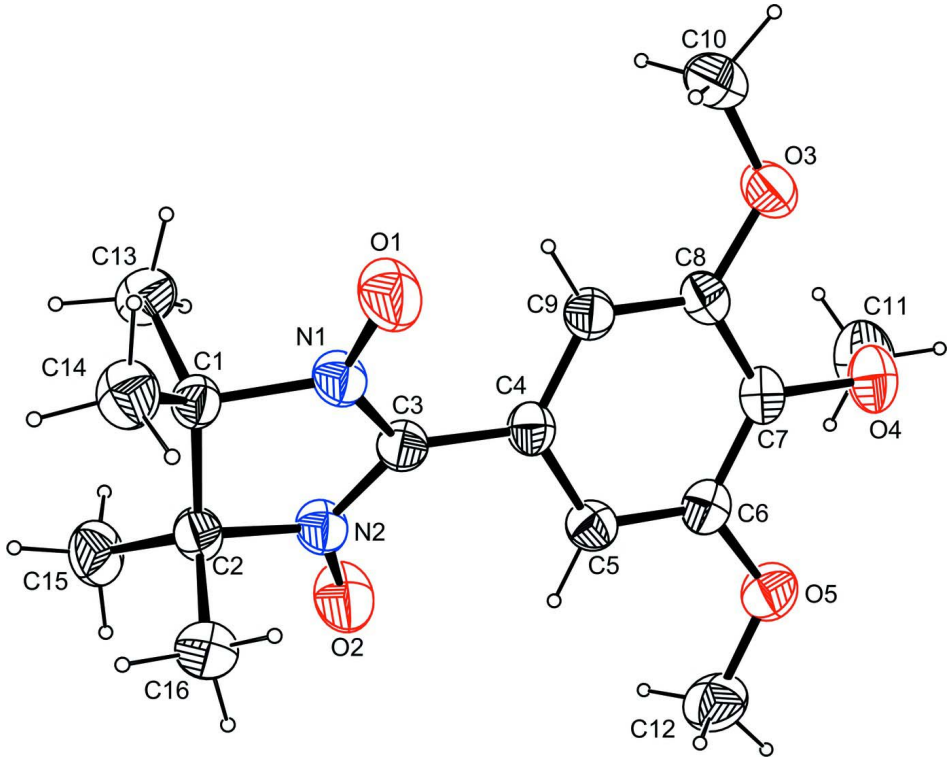



**Figure 1**

Molecular structure of the title compound (I), showing the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

### 4,4,5,5-Tetramethyl-2-(3,4,5-trimethoxyphenyl)imidazolidine-1-oxyl 3-oxide

*Crystal data*

$C_{16}H_{23}N_2O_5$
$M_r = 323.36$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 20.623\ (3)$ Å
$b = 7.2168\ (12)$ Å
$c = 22.831\ (4)$ Å
$V = 3398.0\ (10)$ Å$^3$
$Z = 8$
$F(000) = 1384$
$D_x = 1.264$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1322 reflections
$\theta = 3.1–18.2°$
$\mu = 0.09$ mm$^{-1}$
$T = 296$ K
Block, blue
0.32 × 0.25 × 0.17 mm

*Data collection*

Bruker SMART APEX2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
15858 measured reflections
3027 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.8°$
$h = -12\rightarrow24$
$k = -8\rightarrow8$
$l = -27\rightarrow26$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.119$
$S = 1.06$
3027 reflections
216 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
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{min} = -0.23$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0019 (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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.06793 (9) | 0.2073 (3) | 0.49665 (8) | 0.0570 (5) |
| N2 | 0.15280 (9) | 0.0432 (3) | 0.47376 (8) | 0.0576 (5) |
| O1 | 0.01712 (9) | 0.2652 (3) | 0.52296 (7) | 0.0913 (6) |
| O2 | 0.20243 (8) | −0.0630 (3) | 0.47707 (7) | 0.0903 (6) |
| O3 | 0.07153 (8) | 0.1394 (2) | 0.72841 (6) | 0.0717 (5) |
| O4 | 0.12645 (7) | −0.1894 (2) | 0.74689 (6) | 0.0720 (5) |
| O5 | 0.17240 (8) | −0.3947 (2) | 0.65885 (7) | 0.0743 (5) |
| C1 | 0.08745 (10) | 0.2830 (3) | 0.43780 (9) | 0.0511 (6) |
| C2 | 0.13132 (10) | 0.1227 (3) | 0.41649 (9) | 0.0548 (6) |
| C3 | 0.11082 (10) | 0.0839 (3) | 0.51744 (9) | 0.0506 (6) |
| C4 | 0.11306 (10) | 0.0103 (3) | 0.57690 (9) | 0.0505 (6) |
| C5 | 0.14140 (10) | −0.1615 (3) | 0.58697 (10) | 0.0533 (6) |
| H5 | 0.1574 | −0.2313 | 0.5559 | 0.064* |
| C6 | 0.14551 (10) | −0.2276 (3) | 0.64375 (10) | 0.0550 (6) |
| C7 | 0.12210 (10) | −0.1231 (3) | 0.69035 (10) | 0.0542 (6) |
| C8 | 0.09320 (11) | 0.0479 (3) | 0.67984 (9) | 0.0543 (6) |
| C9 | 0.08840 (10) | 0.1132 (3) | 0.62324 (9) | 0.0539 (6) |
| H9 | 0.0686 | 0.2266 | 0.6161 | 0.065* |
| C10 | 0.03918 (13) | 0.3130 (4) | 0.71996 (10) | 0.0823 (8) |
| H10A | 0.0693 | 0.4022 | 0.7047 | 0.123* |
| H10B | 0.0224 | 0.3562 | 0.7567 | 0.123* |
| H10C | 0.0041 | 0.2971 | 0.6928 | 0.123* |
| C11 | 0.18747 (13) | −0.1598 (5) | 0.77318 (11) | 0.1017 (10) |

| | | | | |
|---|---|---|---|---|
| H11A | 0.2203 | −0.2234 | 0.7511 | 0.153* |
| H11B | 0.1868 | −0.2064 | 0.8126 | 0.153* |
| H11C | 0.1968 | −0.0295 | 0.7737 | 0.153* |
| C12 | 0.19952 (13) | −0.5035 (4) | 0.61337 (12) | 0.0869 (8) |
| H12A | 0.1663 | −0.5354 | 0.5857 | 0.130* |
| H12B | 0.2177 | −0.6146 | 0.6296 | 0.130* |
| H12C | 0.2329 | −0.4343 | 0.5940 | 0.130* |
| C13 | 0.12386 (12) | 0.4629 (3) | 0.45043 (11) | 0.0726 (7) |
| H13A | 0.0965 | 0.5449 | 0.4724 | 0.109* |
| H13B | 0.1358 | 0.5209 | 0.4141 | 0.109* |
| H13C | 0.1622 | 0.4360 | 0.4727 | 0.109* |
| C14 | 0.02814 (11) | 0.3224 (3) | 0.40075 (10) | 0.0722 (7) |
| H14A | 0.0019 | 0.2129 | 0.3984 | 0.108* |
| H14B | 0.0414 | 0.3586 | 0.3621 | 0.108* |
| H14C | 0.0035 | 0.4207 | 0.4183 | 0.108* |
| C15 | 0.18983 (12) | 0.1816 (4) | 0.38065 (10) | 0.0783 (8) |
| H15A | 0.2168 | 0.2619 | 0.4037 | 0.117* |
| H15B | 0.1756 | 0.2462 | 0.3462 | 0.117* |
| H15C | 0.2141 | 0.0739 | 0.3694 | 0.117* |
| C16 | 0.09402 (13) | −0.0316 (3) | 0.38572 (10) | 0.0765 (8) |
| H16A | 0.1214 | −0.1385 | 0.3818 | 0.115* |
| H16B | 0.0808 | 0.0099 | 0.3476 | 0.115* |
| H16C | 0.0564 | −0.0633 | 0.4084 | 0.115* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0488 (12) | 0.0639 (13) | 0.0583 (12) | 0.0123 (10) | 0.0125 (10) | 0.0033 (10) |
| N2 | 0.0509 (11) | 0.0636 (13) | 0.0583 (13) | 0.0127 (11) | 0.0095 (10) | 0.0047 (10) |
| O1 | 0.0890 (14) | 0.1057 (15) | 0.0792 (11) | 0.0366 (11) | 0.0233 (11) | 0.0175 (10) |
| O2 | 0.0745 (12) | 0.1091 (15) | 0.0872 (12) | 0.0401 (11) | 0.0215 (10) | 0.0192 (10) |
| O3 | 0.0827 (13) | 0.0798 (12) | 0.0526 (10) | 0.0157 (10) | 0.0074 (8) | −0.0006 (9) |
| O4 | 0.0655 (12) | 0.0913 (13) | 0.0591 (11) | −0.0002 (9) | −0.0019 (9) | 0.0204 (9) |
| O5 | 0.0798 (12) | 0.0648 (12) | 0.0783 (11) | 0.0196 (10) | 0.0001 (10) | 0.0135 (10) |
| C1 | 0.0527 (14) | 0.0518 (15) | 0.0487 (13) | 0.0028 (12) | 0.0001 (11) | 0.0055 (11) |
| C2 | 0.0590 (15) | 0.0580 (15) | 0.0474 (13) | 0.0022 (12) | 0.0058 (11) | 0.0036 (12) |
| C3 | 0.0454 (13) | 0.0516 (14) | 0.0548 (14) | 0.0060 (12) | 0.0035 (12) | 0.0021 (11) |
| C4 | 0.0450 (13) | 0.0560 (15) | 0.0504 (14) | −0.0007 (12) | 0.0011 (11) | 0.0049 (12) |
| C5 | 0.0475 (14) | 0.0561 (16) | 0.0561 (15) | 0.0046 (11) | 0.0013 (11) | −0.0007 (12) |
| C6 | 0.0453 (14) | 0.0575 (16) | 0.0623 (16) | 0.0001 (12) | −0.0007 (12) | 0.0082 (13) |
| C7 | 0.0478 (14) | 0.0651 (17) | 0.0496 (14) | −0.0037 (13) | 0.0000 (11) | 0.0112 (13) |
| C8 | 0.0517 (14) | 0.0636 (16) | 0.0474 (15) | −0.0029 (13) | 0.0051 (11) | −0.0016 (12) |
| C9 | 0.0533 (14) | 0.0544 (14) | 0.0539 (15) | 0.0038 (12) | 0.0032 (11) | 0.0032 (12) |
| C10 | 0.096 (2) | 0.081 (2) | 0.0701 (16) | 0.0165 (17) | 0.0143 (15) | −0.0100 (15) |
| C11 | 0.089 (2) | 0.144 (3) | 0.0727 (17) | −0.012 (2) | −0.0241 (17) | 0.0182 (18) |
| C12 | 0.087 (2) | 0.0655 (18) | 0.108 (2) | 0.0200 (15) | 0.0072 (18) | 0.0042 (16) |
| C13 | 0.0812 (19) | 0.0569 (16) | 0.0797 (17) | −0.0028 (14) | 0.0029 (14) | 0.0004 (13) |
| C14 | 0.0669 (17) | 0.0819 (19) | 0.0679 (15) | 0.0090 (14) | −0.0072 (13) | 0.0070 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0740 (19) | 0.087 (2) | 0.0736 (16) | 0.0027 (15) | 0.0289 (14) | 0.0119 (14) |
| C16 | 0.097 (2) | 0.0662 (17) | 0.0662 (16) | −0.0037 (15) | −0.0008 (15) | −0.0077 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—O1 | 1.278 (2) | C8—C9 | 1.379 (3) |
| N1—C3 | 1.342 (2) | C9—H9 | 0.9300 |
| N1—C1 | 1.506 (3) | C10—H10A | 0.9600 |
| N2—O2 | 1.281 (2) | C10—H10B | 0.9600 |
| N2—C3 | 1.353 (2) | C10—H10C | 0.9600 |
| N2—C2 | 1.495 (3) | C11—H11A | 0.9600 |
| O3—C8 | 1.366 (2) | C11—H11B | 0.9600 |
| O3—C10 | 1.433 (3) | C11—H11C | 0.9600 |
| O4—C7 | 1.380 (2) | C12—H12A | 0.9600 |
| O4—C11 | 1.410 (3) | C12—H12B | 0.9600 |
| O5—C6 | 1.372 (3) | C12—H12C | 0.9600 |
| O5—C12 | 1.417 (3) | C13—H13A | 0.9600 |
| C1—C14 | 1.514 (3) | C13—H13B | 0.9600 |
| C1—C13 | 1.527 (3) | C13—H13C | 0.9600 |
| C1—C2 | 1.547 (3) | C14—H14A | 0.9600 |
| C2—C15 | 1.519 (3) | C14—H14B | 0.9600 |
| C2—C16 | 1.525 (3) | C14—H14C | 0.9600 |
| C3—C4 | 1.459 (3) | C15—H15A | 0.9600 |
| C4—C9 | 1.389 (3) | C15—H15B | 0.9600 |
| C4—C5 | 1.390 (3) | C15—H15C | 0.9600 |
| C5—C6 | 1.384 (3) | C16—H16A | 0.9600 |
| C5—H5 | 0.9300 | C16—H16B | 0.9600 |
| C6—C7 | 1.391 (3) | C16—H16C | 0.9600 |
| C7—C8 | 1.391 (3) | | |

| | | | |
|---|---|---|---|
| O1—N1—C3 | 126.26 (18) | O3—C10—H10A | 109.5 |
| O1—N1—C1 | 121.31 (18) | O3—C10—H10B | 109.5 |
| C3—N1—C1 | 112.36 (17) | H10A—C10—H10B | 109.5 |
| O2—N2—C3 | 126.70 (19) | O3—C10—H10C | 109.5 |
| O2—N2—C2 | 121.20 (17) | H10A—C10—H10C | 109.5 |
| C3—N2—C2 | 111.83 (17) | H10B—C10—H10C | 109.5 |
| C8—O3—C10 | 117.76 (17) | O4—C11—H11A | 109.5 |
| C7—O4—C11 | 113.82 (18) | O4—C11—H11B | 109.5 |
| C6—O5—C12 | 117.56 (18) | H11A—C11—H11B | 109.5 |
| N1—C1—C14 | 110.53 (17) | O4—C11—H11C | 109.5 |
| N1—C1—C13 | 105.77 (17) | H11A—C11—H11C | 109.5 |
| C14—C1—C13 | 110.08 (19) | H11B—C11—H11C | 109.5 |
| N1—C1—C2 | 99.52 (16) | O5—C12—H12A | 109.5 |
| C14—C1—C2 | 115.92 (18) | O5—C12—H12B | 109.5 |
| C13—C1—C2 | 114.05 (18) | H12A—C12—H12B | 109.5 |
| N2—C2—C15 | 110.07 (18) | O5—C12—H12C | 109.5 |
| N2—C2—C16 | 105.78 (18) | H12A—C12—H12C | 109.5 |
| C15—C2—C16 | 110.91 (19) | H12B—C12—H12C | 109.5 |

| | | | |
|---|---|---|---|
| N2—C2—C1 | 100.67 (16) | C1—C13—H13A | 109.5 |
| C15—C2—C1 | 115.15 (19) | C1—C13—H13B | 109.5 |
| C16—C2—C1 | 113.33 (18) | H13A—C13—H13B | 109.5 |
| N1—C3—N2 | 107.77 (18) | C1—C13—H13C | 109.5 |
| N1—C3—C4 | 126.3 (2) | H13A—C13—H13C | 109.5 |
| N2—C3—C4 | 125.9 (2) | H13B—C13—H13C | 109.5 |
| C9—C4—C5 | 120.32 (19) | C1—C14—H14A | 109.5 |
| C9—C4—C3 | 120.2 (2) | C1—C14—H14B | 109.5 |
| C5—C4—C3 | 119.5 (2) | H14A—C14—H14B | 109.5 |
| C6—C5—C4 | 119.2 (2) | C1—C14—H14C | 109.5 |
| C6—C5—H5 | 120.4 | H14A—C14—H14C | 109.5 |
| C4—C5—H5 | 120.4 | H14B—C14—H14C | 109.5 |
| O5—C6—C5 | 124.3 (2) | C2—C15—H15A | 109.5 |
| O5—C6—C7 | 115.1 (2) | C2—C15—H15B | 109.5 |
| C5—C6—C7 | 120.6 (2) | H15A—C15—H15B | 109.5 |
| O4—C7—C6 | 120.4 (2) | C2—C15—H15C | 109.5 |
| O4—C7—C8 | 119.8 (2) | H15A—C15—H15C | 109.5 |
| C6—C7—C8 | 119.9 (2) | H15B—C15—H15C | 109.5 |
| O3—C8—C9 | 124.9 (2) | C2—C16—H16A | 109.5 |
| O3—C8—C7 | 115.40 (19) | C2—C16—H16B | 109.5 |
| C9—C8—C7 | 119.7 (2) | H16A—C16—H16B | 109.5 |
| C8—C9—C4 | 120.3 (2) | C2—C16—H16C | 109.5 |
| C8—C9—H9 | 119.8 | H16A—C16—H16C | 109.5 |
| C4—C9—H9 | 119.8 | H16B—C16—H16C | 109.5 |

| | | | |
|---|---|---|---|
| O1—N1—C1—C14 | −36.5 (3) | C2—N2—C3—C4 | 172.4 (2) |
| C3—N1—C1—C14 | 146.39 (19) | N1—C3—C4—C9 | −26.4 (3) |
| O1—N1—C1—C13 | 82.7 (2) | N2—C3—C4—C9 | 151.8 (2) |
| C3—N1—C1—C13 | −94.5 (2) | N1—C3—C4—C5 | 155.1 (2) |
| O1—N1—C1—C2 | −158.87 (19) | N2—C3—C4—C5 | −26.7 (3) |
| C3—N1—C1—C2 | 24.0 (2) | C9—C4—C5—C6 | −0.9 (3) |
| O2—N2—C2—C15 | −40.2 (3) | C3—C4—C5—C6 | 177.65 (19) |
| C3—N2—C2—C15 | 145.36 (19) | C12—O5—C6—C5 | 2.1 (3) |
| O2—N2—C2—C16 | 79.7 (2) | C12—O5—C6—C7 | −177.4 (2) |
| C3—N2—C2—C16 | −94.8 (2) | C4—C5—C6—O5 | −179.98 (19) |
| O2—N2—C2—C1 | −162.2 (2) | C4—C5—C6—C7 | −0.5 (3) |
| C3—N2—C2—C1 | 23.4 (2) | C11—O4—C7—C6 | 82.3 (3) |
| N1—C1—C2—N2 | −25.95 (19) | C11—O4—C7—C8 | −98.7 (3) |
| C14—C1—C2—N2 | −144.43 (19) | O5—C6—C7—O4 | −0.3 (3) |
| C13—C1—C2—N2 | 86.2 (2) | C5—C6—C7—O4 | −179.84 (19) |
| N1—C1—C2—C15 | −144.27 (19) | O5—C6—C7—C8 | −179.30 (19) |
| C14—C1—C2—C15 | 97.3 (2) | C5—C6—C7—C8 | 1.2 (3) |
| C13—C1—C2—C15 | −32.1 (3) | C10—O3—C8—C9 | 2.5 (3) |
| N1—C1—C2—C16 | 86.5 (2) | C10—O3—C8—C7 | −177.7 (2) |
| C14—C1—C2—C16 | −31.9 (3) | O4—C7—C8—O3 | 0.7 (3) |
| C13—C1—C2—C16 | −161.33 (18) | C6—C7—C8—O3 | 179.72 (19) |
| O1—N1—C3—N2 | 172.6 (2) | O4—C7—C8—C9 | −179.5 (2) |
| C1—N1—C3—N2 | −10.4 (2) | C6—C7—C8—C9 | −0.5 (3) |

| | | | |
|---|---|---|---|
| O1—N1—C3—C4 | −8.9 (4) | O3—C8—C9—C4 | 178.9 (2) |
| C1—N1—C3—C4 | 168.07 (19) | C7—C8—C9—C4 | −0.9 (3) |
| O2—N2—C3—N1 | 176.8 (2) | C5—C4—C9—C8 | 1.6 (3) |
| C2—N2—C3—N1 | −9.1 (2) | C3—C4—C9—C8 | −176.9 (2) |
| O2—N2—C3—C4 | −1.7 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14*A*···*Cg*2$^{i}$ | 0.96 | 2.80 | 3.644 (2) | 147 |

Symmetry code: (i) −*x*, −*y*, −*z*+1.