Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

# 2-[1-(*tert*-Butoxycarbonyl)pyrrolidin-2-yl]-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazole-1-oxyl 3-oxide

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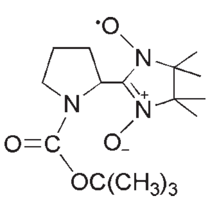
Received 13 May 2010; accepted 31 May 2010

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.042; $wR$ factor = 0.104; data-to-parameter ratio = 7.8.

In the title compound, $C_{16}H_{28}N_3O_4$, the plane of the pyrrolidine ring system is twisted with respect to the plane of the nitronyl nitroxide unit, making a dihedral angle of 79.80 (6)°. The crystal structure is stabilized by C—H⋯O hydrogen bonds.

## Related literature

For the preparation of the title compound, see: Ullman *et al.* (1974). For the properties of nitronyl nitroxide radicals, see: Iqbal *et al.* (2009); Qin *et al.* (2009); Tanaka *et al.* (2007); Soule *et al.* (2007). For puckering parameters, see: Cremer & Pople (1975).




## Experimental

*Crystal data*

$C_{16}H_{28}N_3O_4$
$M_r$ = 326.41
Monoclinic, $P2_1$
$a$ = 6.1016 (12) Å
$b$ = 10.392 (2) Å
$c$ = 14.488 (3) Å
$\beta$ = 101.312 (3)°
$V$ = 900.8 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.36 × 0.28 × 0.17 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
4494 measured reflections
1686 independent reflections
1347 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.048

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.104
$S$ = 0.97
1686 reflections
215 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C2—H2$A$⋯O1 | 0.96 | 2.47 | 3.043 (5) | 118 |
| C3—H3$C$⋯O1 | 0.96 | 2.43 | 3.025 (4) | 120 |
| C16—H17$C$⋯O3$^{i}$ | 0.96 | 2.48 | 3.390 (4) | 157 |

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

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) and *PLATON* (Spek,2009).

We thank the Natural Science Foundation of China (grant Nos. 20972189, 20802092, 20802091) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2566).

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# supporting information

*Acta Cryst.* (2010). E**66**, o1954 [https://doi.org/10.1107/S1600536810020672]

## 2-[1-(*tert*-Butoxycarbonyl)pyrrolidin-2-yl]-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazole-1-oxyl 3-oxide

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

### S1. Comment

Nitronyl nitroxide radical is a class of important functionalized molecule, which has characteristics of magnetism, anticancer, antiradiation and antioxidation, *etc* (Iqbal, *et al.*, 2009; Qin, *et al.*, 2009; Tanaka, *et al.*, 2007; Soule, *et al.*, 2007). The title compound has been be used for coordination with many metalcations, such as $Mn^{2+}$, $Cu^{2+}$ and $Ni^{2+}$ leading to form some molecule based magentic materials. The molecular structure of the title compound is shown in Fig1. The pyrrolidine ring and the nitronyl nitroxide ring are twisted with respect to each other making a dihedral angle of 79.80 (6)°. The crystal structure is stabilized by C—H···O hydrogen bonds (Table 1).

### S2. Experimental

2,3-Dimethyl-2,3-bis(hydroxylamino) butane (1.48 g, 10.0 mmol) and *tert*-butyl-2-(hydroxymethyl) pyrrolidine-1-carboxylate (2.01 g, 10.0 mmol) were dissolved in methanol (Ullman, *et al.*, 1974). The reaction was stirred for 15 h at reflux temperature, then cooled to room temperature and filtered. The white powder was washed by methanol and suspended in a mixed solution of dichloromethane (30.0 ml) and water (30.0 ml). Then the reaction mixture was added to an aqueous solution of $NaIO_4$ and stirred for 15 min in ice bath to give a blue solution. The aqueous phase was extracted with $CH_2Cl_2$ and the organic layer was combined and dried over $MgSO_4$. Then the solvent was removed to give a dark red residue which was purified by a flash column chromatography with the elution of *n*-hexane/ ethyl acetate (1:3) 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

In both structures all the H atoms were discernible in the difference Fourier maps. However, they were constrained by riding model approximation. $C—H_{methyl}$=0.96 Å; $C—H_{aryl}$=0.93 Å; $U_{iso}H_{methyl}$ and $U_{iso}$ $H_{aryl}$ are 1.5 $U_{eq}$ (C) and 1.2 $U_{eq}$ (C), respectively.

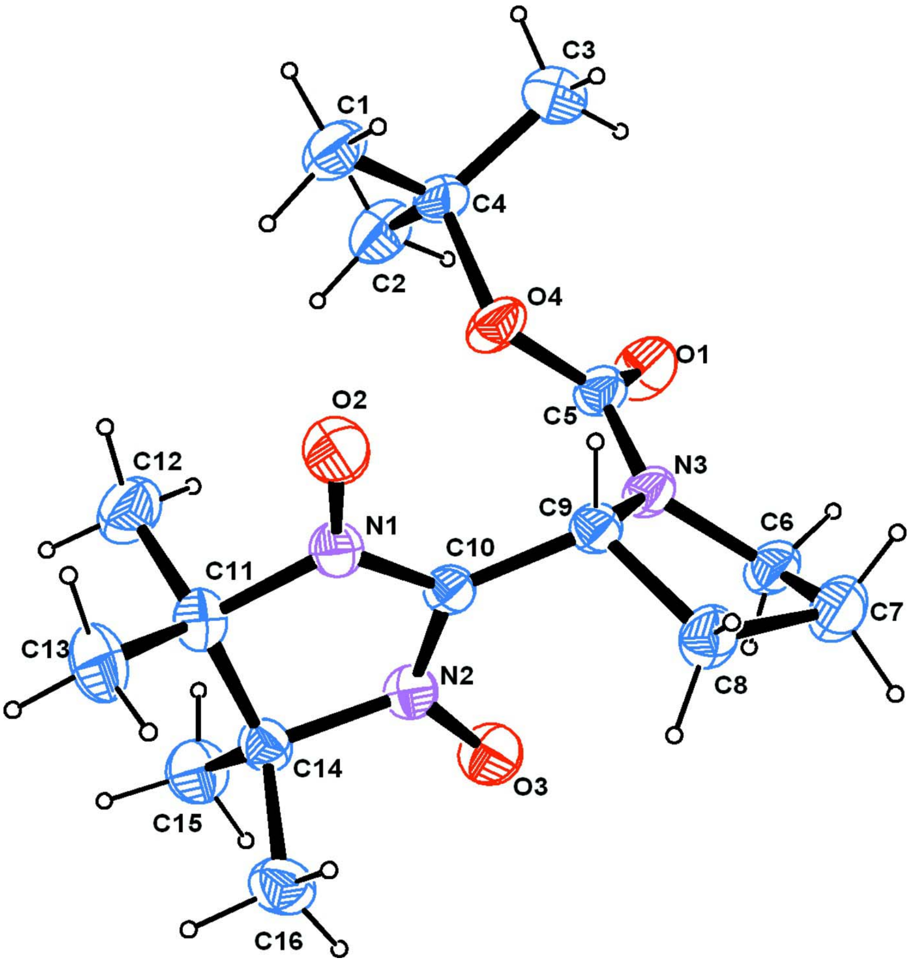


**Figure 1**

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

## 2-[1-(*tert*-Butoxycarbonyl)pyrrolidin-2-yl]-4,4,5,5-tetramethyl-4,5- dihydro-1*H*-imidazole-1-oxyl 3-oxide

### *Crystal data*

$C_{16}H_{28}N_3O_4$
$M_r$ = 326.41
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a$ = 6.1016 (12) Å
$b$ = 10.392 (2) Å
$c$ = 14.488 (3) Å
$\beta$ = 101.312 (3)°
$V$ = 900.8 (3) Å$^3$
$Z$ = 2
$F$(000) = 354
$D_x$ = 1.203 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1546 reflections
$\theta$ = 2.4–21.6°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
Block, red
0.36 × 0.28 × 0.17 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
4494 measured reflections
1686 independent reflections
1347 reflections with $I > 2\sigma(I)$

$R_{int} = 0.048$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.4°$
$h = -7 \rightarrow 7$
$k = -6 \rightarrow 12$
$l = -16 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.104$
$S = 0.97$
1686 reflections
215 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å$^{-3}$
$\Delta\rho_{min} = -0.20$ e Å$^{-3}$
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.022 (5)

*Special details*

**Experimental**. The absolute structure cannot be determined beacuse there are no atoms heaver than silicon in the molecular.

**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 | 0.0929 (4) | 0.3494 (3) | 0.78508 (14) | 0.0442 (6) |
| N2 | 0.3330 (4) | 0.4804 (2) | 0.86782 (15) | 0.0434 (6) |
| N3 | 0.1712 (4) | 0.6757 (2) | 0.71424 (15) | 0.0471 (6) |
| O1 | 0.3991 (4) | 0.7310 (3) | 0.61493 (15) | 0.0669 (7) |
| O2 | −0.0793 (3) | 0.3080 (2) | 0.72759 (14) | 0.0615 (6) |
| O3 | 0.4286 (4) | 0.5857 (2) | 0.90224 (15) | 0.0621 (6) |
| O4 | 0.2361 (4) | 0.5320 (2) | 0.61039 (13) | 0.0575 (6) |
| C1 | 0.2011 (6) | 0.3558 (4) | 0.5093 (2) | 0.0656 (9) |
| H1A | 0.2562 | 0.3044 | 0.5640 | 0.098* |
| H1B | 0.2408 | 0.3160 | 0.4550 | 0.098* |
| H1C | 0.0414 | 0.3625 | 0.5004 | 0.098* |
| C2 | 0.5524 (6) | 0.4831 (4) | 0.5354 (3) | 0.0718 (10) |
| H2A | 0.6115 | 0.5689 | 0.5434 | 0.108* |
| H2B | 0.5934 | 0.4448 | 0.4809 | 0.108* |
| H2C | 0.6123 | 0.4327 | 0.5900 | 0.108* |
| C3 | 0.1978 (7) | 0.5764 (4) | 0.4421 (2) | 0.0799 (12) |
| H3A | 0.0387 | 0.5782 | 0.4375 | 0.120* |
| H3B | 0.2316 | 0.5450 | 0.3842 | 0.120* |

| | | | | |
|---|---|---|---|---|
| H3C | 0.2569 | 0.6617 | 0.4541 | 0.120* |
| C4 | 0.3028 (5) | 0.4878 (3) | 0.52248 (18) | 0.0488 (8) |
| C5 | 0.2803 (5) | 0.6526 (3) | 0.64280 (19) | 0.0482 (7) |
| C6 | 0.1631 (6) | 0.8032 (3) | 0.7549 (2) | 0.0556 (8) |
| H6A | 0.1705 | 0.8699 | 0.7088 | 0.067* |
| H6B | 0.2842 | 0.8153 | 0.8086 | 0.067* |
| C7 | −0.0620 (7) | 0.8035 (3) | 0.7848 (3) | 0.0690 (10) |
| H9A | −0.0623 | 0.8630 | 0.8364 | 0.083* |
| H9B | −0.1810 | 0.8269 | 0.7327 | 0.083* |
| C8 | −0.0877 (6) | 0.6663 (3) | 0.8153 (2) | 0.0576 (9) |
| H10A | −0.2443 | 0.6437 | 0.8084 | 0.069* |
| H10B | −0.0150 | 0.6542 | 0.8806 | 0.069* |
| C9 | 0.0256 (4) | 0.5839 (3) | 0.74985 (18) | 0.0417 (7) |
| H11 | −0.0878 | 0.5518 | 0.6974 | 0.050* |
| C10 | 0.1523 (4) | 0.4731 (3) | 0.79995 (17) | 0.0385 (6) |
| C11 | 0.2665 (5) | 0.2596 (3) | 0.8383 (2) | 0.0506 (8) |
| C12 | 0.4042 (6) | 0.2131 (4) | 0.7676 (2) | 0.0705 (11) |
| H15A | 0.3088 | 0.1683 | 0.7172 | 0.106* |
| H15B | 0.5194 | 0.1560 | 0.7983 | 0.106* |
| H15C | 0.4709 | 0.2856 | 0.7427 | 0.106* |
| C13 | 0.1549 (6) | 0.1467 (4) | 0.8763 (3) | 0.0692 (10) |
| H16A | 0.0599 | 0.1775 | 0.9170 | 0.104* |
| H16B | 0.2669 | 0.0909 | 0.9110 | 0.104* |
| H16C | 0.0670 | 0.0999 | 0.8249 | 0.104* |
| C14 | 0.3947 (4) | 0.3532 (3) | 0.91403 (18) | 0.0450 (7) |
| C15 | 0.6454 (5) | 0.3379 (4) | 0.9356 (2) | 0.0635 (9) |
| H18A | 0.7020 | 0.3464 | 0.8786 | 0.095* |
| H18B | 0.6833 | 0.2544 | 0.9625 | 0.095* |
| H18C | 0.7105 | 0.4031 | 0.9795 | 0.095* |
| C16 | 0.3053 (5) | 0.3565 (4) | 1.00551 (19) | 0.0596 (9) |
| H17A | 0.1457 | 0.3658 | 0.9911 | 0.089* |
| H17B | 0.3702 | 0.4279 | 1.0432 | 0.089* |
| H17C | 0.3439 | 0.2778 | 1.0396 | 0.089* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0490 (13) | 0.0398 (15) | 0.0440 (12) | −0.0025 (12) | 0.0091 (10) | 0.0019 (11) |
| N2 | 0.0451 (13) | 0.0403 (15) | 0.0455 (12) | −0.0073 (13) | 0.0104 (10) | 0.0000 (12) |
| N3 | 0.0717 (15) | 0.0350 (13) | 0.0405 (11) | −0.0039 (13) | 0.0252 (11) | −0.0009 (11) |
| O1 | 0.0936 (17) | 0.0510 (14) | 0.0679 (13) | −0.0193 (14) | 0.0449 (12) | −0.0023 (12) |
| O2 | 0.0602 (12) | 0.0519 (15) | 0.0653 (12) | −0.0125 (11) | −0.0050 (10) | −0.0020 (11) |
| O3 | 0.0670 (13) | 0.0529 (15) | 0.0627 (12) | −0.0176 (13) | 0.0034 (10) | −0.0058 (12) |
| O4 | 0.0907 (15) | 0.0449 (13) | 0.0470 (11) | −0.0064 (13) | 0.0380 (10) | −0.0074 (10) |
| C1 | 0.087 (2) | 0.058 (2) | 0.0559 (17) | −0.002 (2) | 0.0249 (15) | −0.0131 (17) |
| C2 | 0.072 (2) | 0.069 (3) | 0.078 (2) | 0.002 (2) | 0.0212 (16) | −0.020 (2) |
| C3 | 0.113 (3) | 0.077 (3) | 0.0487 (17) | 0.017 (2) | 0.0119 (17) | 0.0065 (19) |
| C4 | 0.0631 (17) | 0.052 (2) | 0.0350 (13) | 0.0042 (16) | 0.0191 (11) | −0.0044 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0683 (19) | 0.0390 (18) | 0.0405 (13) | −0.0040 (16) | 0.0183 (13) | −0.0013 (14) |
| C6 | 0.085 (2) | 0.0382 (17) | 0.0477 (15) | −0.0046 (18) | 0.0227 (15) | −0.0050 (14) |
| C7 | 0.102 (3) | 0.048 (2) | 0.0658 (19) | 0.009 (2) | 0.0381 (18) | 0.0000 (17) |
| C8 | 0.072 (2) | 0.051 (2) | 0.0579 (17) | 0.0066 (18) | 0.0331 (15) | 0.0045 (16) |
| C9 | 0.0506 (15) | 0.0379 (16) | 0.0382 (12) | −0.0024 (14) | 0.0123 (11) | 0.0002 (13) |
| C10 | 0.0457 (14) | 0.0363 (16) | 0.0358 (12) | −0.0047 (14) | 0.0141 (11) | 0.0014 (13) |
| C11 | 0.0507 (17) | 0.043 (2) | 0.0583 (17) | 0.0013 (15) | 0.0113 (13) | 0.0030 (14) |
| C12 | 0.073 (2) | 0.069 (3) | 0.072 (2) | 0.013 (2) | 0.0214 (17) | −0.014 (2) |
| C13 | 0.077 (2) | 0.045 (2) | 0.084 (2) | −0.0033 (19) | 0.0152 (18) | 0.0179 (19) |
| C14 | 0.0482 (15) | 0.0446 (18) | 0.0428 (14) | 0.0008 (15) | 0.0104 (11) | 0.0058 (14) |
| C15 | 0.0514 (16) | 0.071 (3) | 0.0672 (18) | 0.0057 (18) | 0.0081 (14) | 0.0002 (19) |
| C16 | 0.0642 (17) | 0.071 (2) | 0.0466 (15) | 0.0057 (19) | 0.0187 (13) | 0.0097 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—O2 | 1.281 (3) | C6—H6B | 0.9700 |
| N1—C10 | 1.341 (4) | C7—C8 | 1.511 (5) |
| N1—C11 | 1.506 (4) | C7—H9A | 0.9700 |
| N2—O3 | 1.293 (3) | C7—H9B | 0.9700 |
| N2—C10 | 1.328 (3) | C8—C9 | 1.538 (4) |
| N2—C14 | 1.495 (4) | C8—H10A | 0.9700 |
| N3—C5 | 1.357 (4) | C8—H10B | 0.9700 |
| N3—C6 | 1.455 (4) | C9—C10 | 1.494 (4) |
| N3—C9 | 1.464 (4) | C9—H11 | 0.9800 |
| O1—C5 | 1.211 (4) | C11—C13 | 1.513 (5) |
| O4—C5 | 1.347 (4) | C11—C12 | 1.526 (4) |
| O4—C4 | 1.484 (3) | C11—C14 | 1.557 (4) |
| C1—C4 | 1.502 (5) | C12—H15A | 0.9600 |
| C1—H1A | 0.9600 | C12—H15B | 0.9600 |
| C1—H1B | 0.9600 | C12—H15C | 0.9600 |
| C1—H1C | 0.9600 | C13—H16A | 0.9600 |
| C2—C4 | 1.499 (5) | C13—H16B | 0.9600 |
| C2—H2A | 0.9600 | C13—H16C | 0.9600 |
| C2—H2B | 0.9600 | C14—C15 | 1.509 (4) |
| C2—H2C | 0.9600 | C14—C16 | 1.529 (4) |
| C3—C4 | 1.523 (4) | C15—H18A | 0.9600 |
| C3—H3A | 0.9600 | C15—H18B | 0.9600 |
| C3—H3B | 0.9600 | C15—H18C | 0.9600 |
| C3—H3C | 0.9600 | C16—H17A | 0.9600 |
| C6—C7 | 1.518 (5) | C16—H17B | 0.9600 |
| C6—H6A | 0.9700 | C16—H17C | 0.9600 |
| | | | |
| O2—N1—C10 | 126.0 (2) | C7—C8—H10A | 110.7 |
| O2—N1—C11 | 122.0 (3) | C9—C8—H10A | 110.7 |
| C10—N1—C11 | 111.8 (2) | C7—C8—H10B | 110.7 |
| O3—N2—C10 | 125.4 (2) | C9—C8—H10B | 110.7 |
| O3—N2—C14 | 121.35 (19) | H10A—C8—H10B | 108.8 |
| C10—N2—C14 | 112.4 (2) | N3—C9—C10 | 112.4 (2) |

| | | | |
|---|---|---|---|
| C5—N3—C6 | 122.0 (3) | N3—C9—C8 | 103.4 (2) |
| C5—N3—C9 | 125.2 (2) | C10—C9—C8 | 112.4 (2) |
| C6—N3—C9 | 112.3 (2) | N3—C9—H11 | 109.5 |
| C5—O4—C4 | 121.2 (2) | C10—C9—H11 | 109.5 |
| C4—C1—H1A | 109.5 | C8—C9—H11 | 109.5 |
| C4—C1—H1B | 109.5 | N2—C10—N1 | 109.4 (2) |
| H1A—C1—H1B | 109.5 | N2—C10—C9 | 126.2 (3) |
| C4—C1—H1C | 109.5 | N1—C10—C9 | 124.3 (2) |
| H1A—C1—H1C | 109.5 | N1—C11—C13 | 110.2 (2) |
| H1B—C1—H1C | 109.5 | N1—C11—C12 | 106.1 (2) |
| C4—C2—H2A | 109.5 | C13—C11—C12 | 110.2 (3) |
| C4—C2—H2B | 109.5 | N1—C11—C14 | 100.3 (2) |
| H2A—C2—H2B | 109.5 | C13—C11—C14 | 115.4 (3) |
| C4—C2—H2C | 109.5 | C12—C11—C14 | 113.9 (2) |
| H2A—C2—H2C | 109.5 | C11—C12—H15A | 109.5 |
| H2B—C2—H2C | 109.5 | C11—C12—H15B | 109.5 |
| C4—C3—H3A | 109.5 | H15A—C12—H15B | 109.5 |
| C4—C3—H3B | 109.5 | C11—C12—H15C | 109.5 |
| H3A—C3—H3B | 109.5 | H15A—C12—H15C | 109.5 |
| C4—C3—H3C | 109.5 | H15B—C12—H15C | 109.5 |
| H3A—C3—H3C | 109.5 | C11—C13—H16A | 109.5 |
| H3B—C3—H3C | 109.5 | C11—C13—H16B | 109.5 |
| O4—C4—C2 | 110.3 (2) | H16A—C13—H16B | 109.5 |
| O4—C4—C1 | 102.5 (2) | C11—C13—H16C | 109.5 |
| C2—C4—C1 | 111.7 (3) | H16A—C13—H16C | 109.5 |
| O4—C4—C3 | 108.9 (3) | H16B—C13—H16C | 109.5 |
| C2—C4—C3 | 112.3 (3) | N2—C14—C15 | 110.0 (3) |
| C1—C4—C3 | 110.7 (3) | N2—C14—C16 | 105.5 (3) |
| O1—C5—O4 | 127.0 (3) | C15—C14—C16 | 110.0 (2) |
| O1—C5—N3 | 123.4 (3) | N2—C14—C11 | 100.9 (2) |
| O4—C5—N3 | 109.6 (3) | C15—C14—C11 | 115.4 (3) |
| N3—C6—C7 | 102.8 (3) | C16—C14—C11 | 114.1 (2) |
| N3—C6—H6A | 111.2 | C14—C15—H18A | 109.5 |
| C7—C6—H6A | 111.2 | C14—C15—H18B | 109.5 |
| N3—C6—H6B | 111.2 | H18A—C15—H18B | 109.5 |
| C7—C6—H6B | 111.2 | C14—C15—H18C | 109.5 |
| H6A—C6—H6B | 109.1 | H18A—C15—H18C | 109.5 |
| C8—C7—C6 | 103.5 (3) | H18B—C15—H18C | 109.5 |
| C8—C7—H9A | 111.1 | C14—C16—H17A | 109.5 |
| C6—C7—H9A | 111.1 | C14—C16—H17B | 109.5 |
| C8—C7—H9B | 111.1 | H17A—C16—H17B | 109.5 |
| C6—C7—H9B | 111.1 | C14—C16—H17C | 109.5 |
| H9A—C7—H9B | 109.0 | H17A—C16—H17C | 109.5 |
| C7—C8—C9 | 105.0 (2) | H17B—C16—H17C | 109.5 |

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| C5—O4—C4—C2 | 66.4 (4) | C11—N1—C10—C9 | 173.9 (2) |
| C5—O4—C4—C1 | −174.5 (3) | N3—C9—C10—N2 | 50.4 (3) |
| C5—O4—C4—C3 | −57.3 (4) | C8—C9—C10—N2 | −65.8 (4) |

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| C4—O4—C5—O1 | −10.5 (5) | N3—C9—C10—N1 | −132.5 (3) |
| C4—O4—C5—N3 | 169.9 (2) | C8—C9—C10—N1 | 111.3 (3) |
| C6—N3—C5—O1 | 8.7 (5) | O2—N1—C11—C13 | −43.4 (4) |
| C9—N3—C5—O1 | 179.8 (3) | C10—N1—C11—C13 | 141.6 (3) |
| C6—N3—C5—O4 | −171.7 (3) | O2—N1—C11—C12 | 75.8 (3) |
| C9—N3—C5—O4 | −0.7 (4) | C10—N1—C11—C12 | −99.2 (3) |
| C5—N3—C6—C7 | 148.7 (3) | O2—N1—C11—C14 | −165.5 (2) |
| C9—N3—C6—C7 | −23.4 (3) | C10—N1—C11—C14 | 19.5 (3) |
| N3—C6—C7—C8 | 34.8 (3) | O3—N2—C14—C15 | −48.6 (3) |
| C6—C7—C8—C9 | −34.1 (3) | C10—N2—C14—C15 | 141.3 (2) |
| C5—N3—C9—C10 | 69.1 (3) | O3—N2—C14—C16 | 70.0 (3) |
| C6—N3—C9—C10 | −119.1 (3) | C10—N2—C14—C16 | −100.1 (3) |
| C5—N3—C9—C8 | −169.4 (3) | O3—N2—C14—C11 | −171.0 (2) |
| C6—N3—C9—C8 | 2.4 (3) | C10—N2—C14—C11 | 19.0 (3) |
| C7—C8—C9—N3 | 19.9 (3) | N1—C11—C14—N2 | −21.2 (2) |
| C7—C8—C9—C10 | 141.4 (3) | C13—C11—C14—N2 | −139.5 (3) |
| O3—N2—C10—N1 | −177.0 (2) | C12—C11—C14—N2 | 91.6 (3) |
| C14—N2—C10—N1 | −7.4 (3) | N1—C11—C14—C15 | −139.7 (3) |
| O3—N2—C10—C9 | 0.5 (4) | C13—C11—C14—C15 | 102.0 (3) |
| C14—N2—C10—C9 | 170.0 (2) | C12—C11—C14—C15 | −26.8 (4) |
| O2—N1—C10—N2 | 176.7 (2) | N1—C11—C14—C16 | 91.4 (3) |
| C11—N1—C10—N2 | −8.6 (3) | C13—C11—C14—C16 | −26.9 (4) |
| O2—N1—C10—C9 | −0.8 (4) | C12—C11—C14—C16 | −155.7 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···O1 | 0.96 | 2.47 | 3.043 (5) | 118 |
| C3—H3*C*···O1 | 0.96 | 2.43 | 3.025 (4) | 120 |
| C9—H11···O2 | 0.98 | 2.57 | 2.942 (4) | 102 |
| C16—H17*C*···O3$^{i}$ | 0.96 | 2.48 | 3.390 (4) | 157 |

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