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

# Methyl 2-{6-[(1-methoxy-1-oxopropan-2-yl)aminocarbonyl]pyridine-2-carboxamido}propanoate

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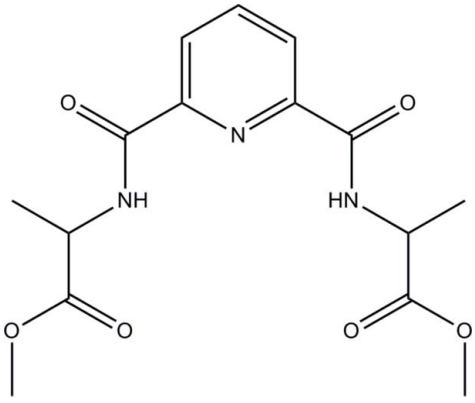
Received 6 May 2012; accepted 16 May 2012

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.053; $wR$ factor = 0.192; data-to-parameter ratio = 11.8.

In the title compound, $C_{15}H_{19}N_3O_6$, the amide planes are inclined at dihedral angles of 0.8 (6) and 12.1 (3)° with respect to the central pyridine ring. The mean planes of the corresponding methyl acetate groups form dihedral angles of 41.76 (13) and 86.48 (15)°, respectively with the mean plane of pyridine ring. A pair of weak intramolecular N—H···N hydrogen bonds generate an $S$(5)$S$(5) ring motif in the molecule. In the crystal, molecules are linked by N—H···O hydrogen bonds into [001] chains. The chains are cross-linked by C—H···O hydrogen bonds into layers lying parallel to $bc$ plane. The crystal packing also features a C—H···$\pi$ interaction.

## Related literature

For the synthesis and biological activity screening of some dipicolinic acid bis-L-amino acid hydrazide derivatives and their corresponding acids, see: Abou-Ghalia & Amr (2004); Al-Salahi *et al.* (2010); Al-Omar & Amr (2010); Attia *et al.* (2000). For the biological activity of 2,6-disubstituted pyridine derivatives, see: Amr (2005); Abou-Ghalia *et al.* (2003); Amr, Sayed & Abdulla (2005); Amr *et al.* (2006); Hammam *et al.* (2003). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{15}H_{19}N_3O_6$
$M_r$ = 337.33
Monoclinic, $P2_1/c$
$a$ = 8.9735 (3) Å
$b$ = 20.7073 (8) Å
$c$ = 10.4048 (5) Å
$\beta$ = 122.901 (3)°
$V$ = 1623.29 (11) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.91 mm$^{-1}$
$T$ = 296 K
0.74 × 0.25 × 0.06 mm

### Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.551, $T_{max}$ = 0.947
10358 measured reflections
2703 independent reflections
2058 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.192
$S$ = 1.04
2703 reflections
229 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

*Cg*1 is the centroid of the N1/C1–C5 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H1*N3*···N1 | 0.85 (3) | 2.23 (3) | 2.676 (3) | 113 (2) |
| N3—H1*N3*···O4$^{i}$ | 0.85 (2) | 2.35 (2) | 3.080 (2) | 145 (2) |
| N2—H1*N2*···N1 | 0.84 (3) | 2.32 (3) | 2.685 (3) | 107 (3) |
| N2—H1*N2*···O4$^{i}$ | 0.83 (3) | 2.55 (3) | 3.290 (3) | 149 (2) |
| C9—H9*B*···O2$^{ii}$ | 0.96 | 2.41 | 3.329 (3) | 159 |
| C15—H15*B*···*Cg*1$^{iii}$ | 0.96 | 2.78 | 3.544 (4) | 137 |

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

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).

MAO, AEA and HAG thank the Deanship of Scientific Research at King Saud University for funding through the research group project No. RGP-VPP-099. HKF and TSC

‡ Thomson Reuters ResearcherID: A-3561-2009.

thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). TSC also thanks the Malaysian Government and USM for the award of a research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6780).

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# supporting information

*Acta Cryst.* (2012). E**68**, o1837–o1838 [doi:10.1107/S1600536812022258]

# Methyl 2-{6-[(1-methoxy-1-oxopropan-2-yl)aminocarbonyl]pyridine-2-carboxamido}propanoate

**Mohamed A. Al-Omar, Abdel-Galil E. Amr, Hazem A. Ghabbour, Tze Shyang Chia and Hoong-Kun Fun**

## S1. Comment

In our previous work (Abou-Ghalia & Amr, 2004; Al-Salahi *et al.*, 2010; Al-Omar & Amr, 2010), we have reported the synthesis and biological activity screening of some dipicolinic acid bis-*L*-amino acid hydrazide derivatives and their corresponding acids (Attia *et al.*, 2000). In view of the significance of 2,6-disubstituted pyridine derivatives as biologically active congeners (Amr, 2005; Abou-Ghalia, Amr & Abdulla, 2003; Amr, Sayed & Abdulla, 2005; Amr *et al.*, 2006; Hammam *et al.*, 2003), we report herein the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound is shown in Fig. 1. The amide planes (O1/N2/C6 & O4/N3/C11) are inclined at dihedral angles of 0.8 (6) and 12.1 (3)°, respectively, with respect to the central pyridine ring (N1/C1–C5). The mean planes of the methyl acetate groups (O2/O3/C7–C9 with maximum deviation = 0.007 (2) Å at atom O3 & O5/O6/C12–C14 with maximum deviation = 0.011 (2) Å at atom O6) form dihedral angles of 41.76 (13) and 86.48 (15)°, respectively with the mean plane of pyridine ring. Weak intramolecular N2—H1N2···N1 and N3—H1N3···N1 hydrogen bonds (Table 1) generate an S(5)S(5) ring motif (Bernstein *et al.*, 1995) in the molecule.

In the crystal (Fig. 2), molecules are linked by intermolecular N3—H1N3···O4, N2—H1N2···O4 and C9—H9B···O2 hydrogen bonds (Table 1) into two-dimensional networks parallel to *bc* plane. The crystal packing is further stabilized by C—H···$\pi$ interaction (Table 1), involving *Cg*1 which is the centroid of N1/C1–C5 ring.

## S2. Experimental

To a cold mixture (-15 °C) of 2,6-pyridine dicarboxylic acid (0.167 g, 1 mmol) in cold dry tetrahydrofuran (100 ml) and ethyl chloroformate (0.216 g, 2 mmol), triethylamine (0.202 g, 2 mmol) was added with stirring. After 10 min, D-alanyl methyl ester (0.206 g, 2 mmol) was then added. The reaction mixture was stirred for 3 h at -15 °C and then 12 h at r.t. The triethylamine hydrochloride formed was filtered off and the solvent was evaporated under reduced pressure. The residue obtained was dissolved in 150 ml dichloromethane, washed with water, 1 N hydrochloric acid, 1 N sodium bicarbonate and finally with water and dried over anhydrous calcium chloride. The solvent was evaporated under reduced pressure to dryness and the obtained solid was crystallized from dichloromethane to give colourless plates of the title compound.

## S3. Refinement

The atoms H1N2 and H1N3 were located in a difference fourier map and refined freely [N—H = 0.83 (3) and 0.85 (2) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93, 0.96 and 0.98 Å] and refined using a riding model with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C). A rotating group model was applied to the methyl groups.

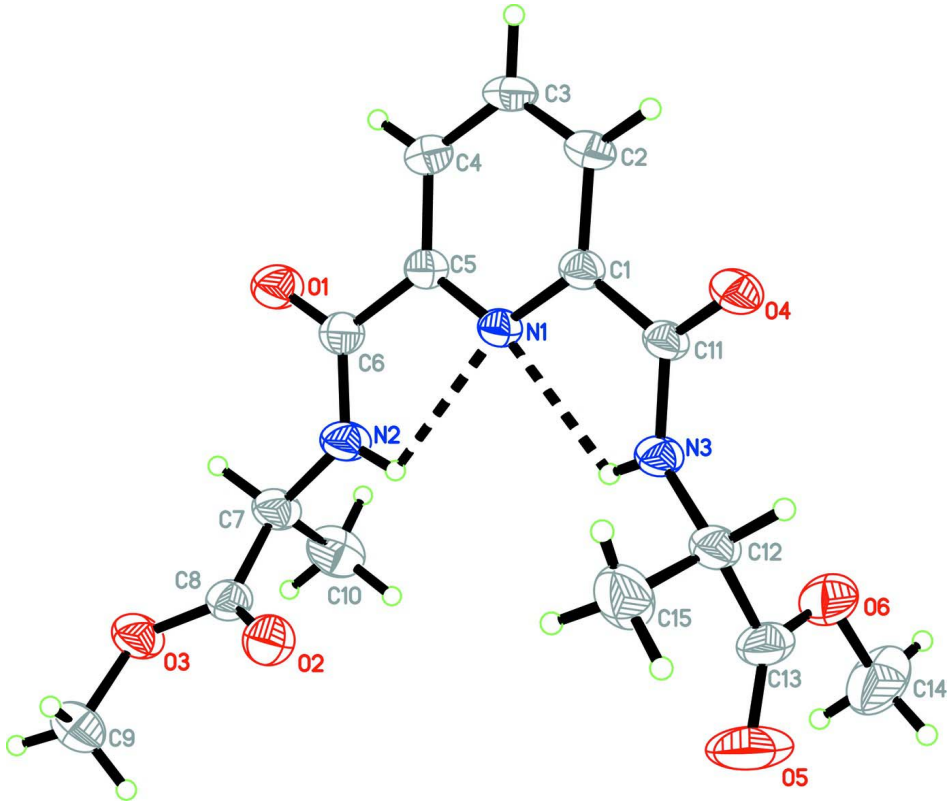


**Figure 1**

The molecular structure of the title compound with 30% probability displacement ellipsoids. The dashed lines represent the weak intramolecular N—H···N hydrogen bonds.

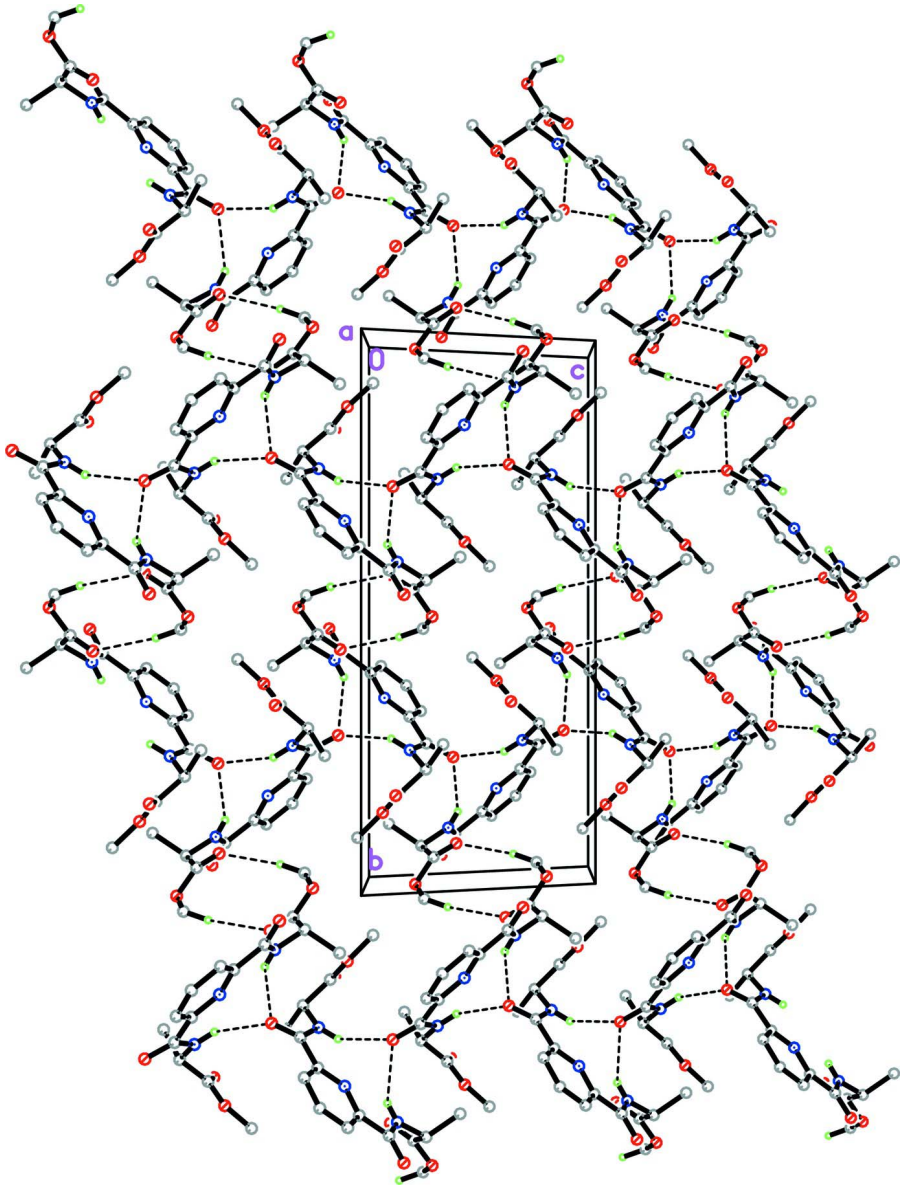


**Figure 2**

The crystal packing of the title compound. The dashed lines represent the hydrogen bonds. For clarity sake, hydrogen atoms not involved in hydrogen bonding have been omitted.

**Methyl 2-{6-[(1-methoxy-1-oxopropan-2-yl)aminocarbonyl]pyridine-2- carboxamido}propanoate**

*Crystal data*

$C_{15}H_{19}N_3O_6$
$M_r$ = 337.33
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 8.9735 (3) Å
$b$ = 20.7073 (8) Å
$c$ = 10.4048 (5) Å
$\beta$ = 122.901 (3)°
$V$ = 1623.29 (11) Å$^3$
$Z$ = 4
$F$(000) = 712
$D_x$ = 1.380 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 1828 reflections
$\theta$ = 5.5–70.4°
$\mu$ = 0.91 mm$^{-1}$

$T$ = 296 K
Plate, colourless

0.74 × 0.25 × 0.06 mm

*Data collection*

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.551, $T_{max}$ = 0.947

10358 measured reflections
2703 independent reflections
2058 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046
$\theta_{max}$ = 65.0°, $\theta_{min}$ = 5.5°
$h$ = −10→10
$k$ = −24→24
$l$ = −11→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.192
$S$ = 1.04
2703 reflections
229 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
$w = 1/[\sigma^2(F_o^2) + (0.1344P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.1466 (3) | 0.53103 (10) | 0.8205 (2) | 0.0711 (6) |
| O2 | 0.4090 (3) | 0.56823 (9) | 0.89076 (19) | 0.0641 (5) |
| O3 | 0.2864 (3) | 0.49181 (10) | 0.7100 (2) | 0.0710 (6) |
| O4 | 0.2803 (2) | 0.77285 (8) | 1.37663 (16) | 0.0578 (5) |
| O5 | 0.6292 (4) | 0.83396 (18) | 1.1329 (3) | 0.1239 (11) |
| O6 | 0.3640 (4) | 0.87058 (11) | 1.0701 (2) | 0.0823 (7) |
| N1 | 0.0899 (2) | 0.66141 (9) | 1.07033 (18) | 0.0438 (5) |
| N2 | 0.0839 (3) | 0.58945 (11) | 0.8529 (2) | 0.0602 (6) |
| N3 | 0.3295 (3) | 0.75674 (9) | 1.1876 (2) | 0.0497 (5) |
| C1 | 0.0965 (3) | 0.69752 (10) | 1.1805 (2) | 0.0449 (5) |
| C2 | −0.0180 (4) | 0.68863 (13) | 1.2288 (3) | 0.0584 (7) |
| H2A | −0.0101 | 0.7145 | 1.3054 | 0.070* |
| C3 | −0.1448 (4) | 0.64055 (14) | 1.1610 (3) | 0.0638 (7) |

| | | | | |
|---|---|---|---|---|
| H3A | −0.2241 | 0.6337 | 1.1910 | 0.077* |
| C4 | −0.1521 (4) | 0.60292 (13) | 1.0484 (3) | 0.0572 (6) |
| H4A | −0.2358 | 0.5701 | 1.0016 | 0.069* |
| C5 | −0.0334 (3) | 0.61483 (11) | 1.0065 (2) | 0.0471 (5) |
| C6 | −0.0374 (3) | 0.57458 (12) | 0.8840 (2) | 0.0530 (6) |
| C7 | 0.0902 (4) | 0.55753 (13) | 0.7325 (3) | 0.0607 (7) |
| H7A | 0.0229 | 0.5172 | 0.7074 | 0.073* |
| C8 | 0.2796 (4) | 0.54102 (12) | 0.7893 (3) | 0.0550 (6) |
| C9 | 0.4596 (4) | 0.46969 (16) | 0.7517 (3) | 0.0766 (8) |
| H9A | 0.4468 | 0.4353 | 0.6845 | 0.115* |
| H9B | 0.5236 | 0.4543 | 0.8553 | 0.115* |
| H9C | 0.5236 | 0.5047 | 0.7430 | 0.115* |
| C10 | 0.0058 (4) | 0.59893 (17) | 0.5880 (3) | 0.0788 (9) |
| H10A | −0.1141 | 0.6092 | 0.5555 | 0.118* |
| H10B | 0.0058 | 0.5755 | 0.5083 | 0.118* |
| H10C | 0.0726 | 0.6381 | 0.6093 | 0.118* |
| C11 | 0.2444 (3) | 0.74632 (10) | 1.2579 (2) | 0.0439 (5) |
| C12 | 0.4982 (3) | 0.79021 (13) | 1.2598 (3) | 0.0566 (6) |
| H12A | 0.5071 | 0.8179 | 1.3401 | 0.068* |
| C13 | 0.5066 (5) | 0.83318 (16) | 1.1474 (3) | 0.0723 (9) |
| C14 | 0.3623 (7) | 0.91492 (19) | 0.9625 (4) | 0.1168 (16) |
| H14A | 0.2584 | 0.9416 | 0.9187 | 0.175* |
| H14B | 0.3610 | 0.8911 | 0.8828 | 0.175* |
| H14C | 0.4665 | 0.9416 | 1.0144 | 0.175* |
| C15 | 0.6504 (4) | 0.74250 (17) | 1.3363 (4) | 0.0899 (10) |
| H15A | 0.6515 | 0.7215 | 1.4191 | 0.135* |
| H15B | 0.7605 | 0.7650 | 1.3753 | 0.135* |
| H15C | 0.6356 | 0.7107 | 1.2630 | 0.135* |
| H1N3 | 0.289 (3) | 0.7353 (11) | 1.105 (3) | 0.044 (6)* |
| H1N2 | 0.151 (4) | 0.6210 (14) | 0.896 (3) | 0.063 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0754 (13) | 0.0729 (12) | 0.0837 (11) | −0.0269 (10) | 0.0553 (11) | −0.0275 (9) |
| O2 | 0.0620 (12) | 0.0660 (11) | 0.0641 (9) | −0.0096 (9) | 0.0342 (9) | −0.0117 (8) |
| O3 | 0.0754 (14) | 0.0750 (12) | 0.0845 (12) | −0.0149 (10) | 0.0576 (11) | −0.0296 (9) |
| O4 | 0.0738 (12) | 0.0660 (10) | 0.0528 (8) | −0.0078 (9) | 0.0469 (9) | −0.0110 (7) |
| O5 | 0.115 (2) | 0.176 (3) | 0.136 (2) | −0.051 (2) | 0.1048 (19) | −0.031 (2) |
| O6 | 0.1178 (18) | 0.0758 (13) | 0.0865 (12) | −0.0121 (13) | 0.0770 (13) | 0.0060 (10) |
| N1 | 0.0479 (11) | 0.0482 (10) | 0.0445 (9) | 0.0006 (8) | 0.0310 (9) | 0.0015 (7) |
| N2 | 0.0646 (14) | 0.0696 (14) | 0.0651 (11) | −0.0223 (12) | 0.0473 (11) | −0.0253 (10) |
| N3 | 0.0573 (13) | 0.0580 (11) | 0.0470 (9) | −0.0132 (9) | 0.0369 (10) | −0.0131 (8) |
| C1 | 0.0506 (13) | 0.0491 (12) | 0.0440 (9) | 0.0040 (10) | 0.0315 (10) | 0.0047 (8) |
| C2 | 0.0720 (17) | 0.0656 (15) | 0.0631 (12) | −0.0023 (13) | 0.0533 (13) | −0.0044 (10) |
| C3 | 0.0662 (16) | 0.0735 (17) | 0.0811 (15) | −0.0105 (14) | 0.0591 (14) | −0.0040 (12) |
| C4 | 0.0602 (16) | 0.0566 (14) | 0.0705 (14) | −0.0088 (12) | 0.0457 (13) | −0.0010 (10) |
| C5 | 0.0490 (13) | 0.0486 (12) | 0.0503 (10) | 0.0000 (10) | 0.0312 (11) | 0.0019 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6 | 0.0542 (15) | 0.0541 (13) | 0.0569 (11) | −0.0060 (11) | 0.0343 (12) | −0.0062 (10) |
| C7 | 0.0639 (16) | 0.0681 (16) | 0.0653 (13) | −0.0184 (13) | 0.0450 (13) | −0.0261 (11) |
| C8 | 0.0679 (17) | 0.0549 (13) | 0.0589 (12) | −0.0104 (13) | 0.0452 (13) | −0.0090 (10) |
| C9 | 0.085 (2) | 0.083 (2) | 0.0836 (17) | 0.0065 (17) | 0.0600 (17) | −0.0092 (14) |
| C10 | 0.072 (2) | 0.105 (2) | 0.0569 (13) | −0.0048 (18) | 0.0335 (15) | −0.0183 (14) |
| C11 | 0.0519 (13) | 0.0489 (12) | 0.0430 (10) | 0.0039 (10) | 0.0337 (10) | 0.0022 (8) |
| C12 | 0.0583 (15) | 0.0647 (15) | 0.0617 (12) | −0.0148 (12) | 0.0422 (12) | −0.0206 (10) |
| C13 | 0.086 (2) | 0.084 (2) | 0.0764 (16) | −0.0386 (18) | 0.0634 (17) | −0.0317 (15) |
| C14 | 0.197 (5) | 0.094 (2) | 0.095 (2) | −0.044 (3) | 0.103 (3) | −0.0047 (18) |
| C15 | 0.0597 (19) | 0.085 (2) | 0.122 (2) | −0.0032 (16) | 0.0471 (18) | −0.0272 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C6 | 1.227 (3) | C4—C5 | 1.375 (3) |
| O2—C8 | 1.204 (3) | C4—H4A | 0.9300 |
| O3—C8 | 1.334 (3) | C5—C6 | 1.507 (3) |
| O3—C9 | 1.442 (4) | C7—C8 | 1.504 (4) |
| O4—C11 | 1.225 (2) | C7—C10 | 1.527 (4) |
| O5—C13 | 1.189 (4) | C7—H7A | 0.9800 |
| O6—C13 | 1.329 (4) | C9—H9A | 0.9600 |
| O6—C14 | 1.441 (3) | C9—H9B | 0.9600 |
| N1—C5 | 1.340 (3) | C9—H9C | 0.9600 |
| N1—C1 | 1.343 (3) | C10—H10A | 0.9600 |
| N2—C6 | 1.329 (3) | C10—H10B | 0.9600 |
| N2—C7 | 1.445 (3) | C10—H10C | 0.9600 |
| N2—H1N2 | 0.83 (3) | C12—C13 | 1.504 (4) |
| N3—C11 | 1.331 (3) | C12—C15 | 1.514 (4) |
| N3—C12 | 1.449 (3) | C12—H12A | 0.9800 |
| N3—H1N3 | 0.85 (2) | C14—H14A | 0.9600 |
| C1—C2 | 1.379 (3) | C14—H14B | 0.9600 |
| C1—C11 | 1.507 (3) | C14—H14C | 0.9600 |
| C2—C3 | 1.383 (4) | C15—H15A | 0.9600 |
| C2—H2A | 0.9300 | C15—H15B | 0.9600 |
| C3—C4 | 1.378 (3) | C15—H15C | 0.9600 |
| C3—H3A | 0.9300 | | |
| | | | |
| C8—O3—C9 | 117.4 (2) | O3—C9—H9A | 109.5 |
| C13—O6—C14 | 116.2 (3) | O3—C9—H9B | 109.5 |
| C5—N1—C1 | 117.75 (19) | H9A—C9—H9B | 109.5 |
| C6—N2—C7 | 121.9 (2) | O3—C9—H9C | 109.5 |
| C6—N2—H1N2 | 119 (2) | H9A—C9—H9C | 109.5 |
| C7—N2—H1N2 | 118 (2) | H9B—C9—H9C | 109.5 |
| C11—N3—C12 | 122.81 (17) | C7—C10—H10A | 109.5 |
| C11—N3—H1N3 | 114.2 (17) | C7—C10—H10B | 109.5 |
| C12—N3—H1N3 | 121.7 (17) | H10A—C10—H10B | 109.5 |
| N1—C1—C2 | 122.7 (2) | C7—C10—H10C | 109.5 |
| N1—C1—C11 | 116.56 (19) | H10A—C10—H10C | 109.5 |
| C2—C1—C11 | 120.61 (19) | H10B—C10—H10C | 109.5 |

| | | | |
|---|---|---|---|
| C1—C2—C3 | 118.7 (2) | O4—C11—N3 | 124.5 (2) |
| C1—C2—H2A | 120.7 | O4—C11—C1 | 120.8 (2) |
| C3—C2—H2A | 120.7 | N3—C11—C1 | 114.65 (17) |
| C4—C3—C2 | 119.1 (2) | N3—C12—C13 | 111.0 (2) |
| C4—C3—H3A | 120.4 | N3—C12—C15 | 110.5 (2) |
| C2—C3—H3A | 120.4 | C13—C12—C15 | 112.5 (3) |
| C5—C4—C3 | 118.7 (2) | N3—C12—H12A | 107.5 |
| C5—C4—H4A | 120.6 | C13—C12—H12A | 107.5 |
| C3—C4—H4A | 120.6 | C15—C12—H12A | 107.5 |
| N1—C5—C4 | 123.0 (2) | O5—C13—O6 | 124.6 (3) |
| N1—C5—C6 | 116.8 (2) | O5—C13—C12 | 123.2 (4) |
| C4—C5—C6 | 120.1 (2) | O6—C13—C12 | 112.2 (2) |
| O1—C6—N2 | 124.0 (2) | O6—C14—H14A | 109.5 |
| O1—C6—C5 | 120.5 (2) | O6—C14—H14B | 109.5 |
| N2—C6—C5 | 115.5 (2) | H14A—C14—H14B | 109.5 |
| N2—C7—C8 | 109.3 (2) | O6—C14—H14C | 109.5 |
| N2—C7—C10 | 111.4 (2) | H14A—C14—H14C | 109.5 |
| C8—C7—C10 | 111.4 (2) | H14B—C14—H14C | 109.5 |
| N2—C7—H7A | 108.2 | C12—C15—H15A | 109.5 |
| C8—C7—H7A | 108.2 | C12—C15—H15B | 109.5 |
| C10—C7—H7A | 108.2 | H15A—C15—H15B | 109.5 |
| O2—C8—O3 | 123.6 (2) | C12—C15—H15C | 109.5 |
| O2—C8—C7 | 125.8 (2) | H15A—C15—H15C | 109.5 |
| O3—C8—C7 | 110.6 (2) | H15B—C15—H15C | 109.5 |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | −0.4 (3) | C9—O3—C8—C7 | −179.5 (2) |
| C5—N1—C1—C11 | 175.80 (18) | N2—C7—C8—O2 | −25.0 (4) |
| N1—C1—C2—C3 | 0.0 (4) | C10—C7—C8—O2 | 98.6 (3) |
| C11—C1—C2—C3 | −176.0 (2) | N2—C7—C8—O3 | 156.0 (2) |
| C1—C2—C3—C4 | 0.4 (4) | C10—C7—C8—O3 | −80.5 (3) |
| C2—C3—C4—C5 | −0.4 (4) | C12—N3—C11—O4 | 13.6 (4) |
| C1—N1—C5—C4 | 0.3 (3) | C12—N3—C11—C1 | −165.8 (2) |
| C1—N1—C5—C6 | −179.55 (19) | N1—C1—C11—O4 | −166.69 (19) |
| C3—C4—C5—N1 | 0.1 (4) | C2—C1—C11—O4 | 9.5 (3) |
| C3—C4—C5—C6 | 179.9 (2) | N1—C1—C11—N3 | 12.8 (3) |
| C7—N2—C6—O1 | 4.1 (4) | C2—C1—C11—N3 | −171.0 (2) |
| C7—N2—C6—C5 | −176.5 (2) | C11—N3—C12—C13 | −141.6 (2) |
| N1—C5—C6—O1 | 180.0 (2) | C11—N3—C12—C15 | 92.8 (3) |
| C4—C5—C6—O1 | 0.1 (4) | C14—O6—C13—O5 | −0.3 (4) |
| N1—C5—C6—N2 | 0.5 (3) | C14—O6—C13—C12 | 178.2 (2) |
| C4—C5—C6—N2 | −179.3 (2) | N3—C12—C13—O5 | −131.9 (3) |
| C6—N2—C7—C8 | −136.0 (3) | C15—C12—C13—O5 | −7.5 (4) |
| C6—N2—C7—C10 | 100.4 (3) | N3—C12—C13—O6 | 49.5 (3) |
| C9—O3—C8—O2 | 1.4 (4) | C15—C12—C13—O6 | 173.9 (2) |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 is the centroid of the N1/C1–C5 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H1*N*3···N1 | 0.85 (3) | 2.23 (3) | 2.676 (3) | 113 (2) |
| N3—H1*N*3···O4$^{i}$ | 0.85 (2) | 2.35 (2) | 3.080 (2) | 145 (2) |
| N2—H1*N*2···N1 | 0.84 (3) | 2.32 (3) | 2.685 (3) | 107 (3) |
| N2—H1*N*2···O4$^{i}$ | 0.83 (3) | 2.55 (3) | 3.290 (3) | 149 (2) |
| C9—H9*B*···O2$^{ii}$ | 0.96 | 2.41 | 3.329 (3) | 159 |
| C15—H15*B*···*Cg*1$^{iii}$ | 0.96 | 2.78 | 3.544 (4) | 137 |

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