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

# (1′*S*,2*R*,3*R*)-(−)-2-Hydroxy-3-morpholino-3-phenyl-*N*-(1′-phenylethyl)propionamide

**Angel Mendoza,* David M. Aparicio, Joel L. Terán, Dino Gnecco and Jorge R. Juárez**

Centro de Química, ICUAP, Benemérita Universidad Autónoma de Puebla, Puebla, Puebla, Mexico
Correspondence e-mail: angel.mendoza.m@gmail.com

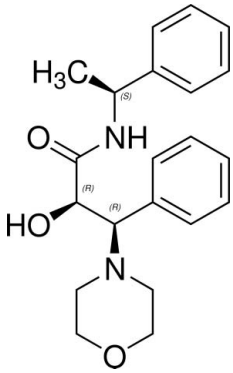
Received 3 March 2009; accepted 5 March 2009

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.054; $wR$ factor = 0.163; data-to-parameter ratio = 11.8.

In the title compound, $C_{21}H_{26}N_2O_3$, the morpholine ring has a chair conformation and the dihedral angle between the two phenyl rings is 59.0 (3)°. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds, generating a ribbon structure along the *a* axis. An intramolecular N—H···O contact is also present.

## Related literature

For general background, see: Barbaro *et al.* (1992); Szymanski *et al.* (2006); Sheppard *et al.* (2004); Chen *et al.* (1996); Concellón *et al.* (2003*a*,*b*); Martín *et al.* (2004). For related structures, see: Romero *et al.* (2005*a*,*b*). For ring conformation analysis, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{21}H_{26}N_2O_3$
$M_r$ = 354.44
Orthorhombic, $P2_12_12_1$
$a$ = 6.0010 (17) Å
$b$ = 15.659 (3) Å
$c$ = 20.746 (4) Å
$V$ = 1949.5 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 K
0.72 × 0.28 × 0.16 mm

### Data collection

Bruker P4 diffractometer
Absorption correction: none
3964 measured reflections
2959 independent reflections
1116 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.055
3 standard reflections every 97 reflections
intensity decay: 3%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.163
$S$ = 0.89
2959 reflections
251 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*···O1 | 1.00 (7) | 1.92 (7) | 2.569 (5) | 120 (5) |
| O1—H1*O*···O2$^{i}$ | 0.98 (8) | 1.80 (8) | 2.753 (4) | 163 (7) |

Symmetry code: (i) $x-1, y, z$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We acknowledge financial support under scholarship DMAS No. 169011 and CONACyT project No. 83185. Special thanks go to Dr Marcos Flores (USAI-FQ-UNAM) for useful comments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2398).

## References

Barbaro, G., Battaglia, A. & Giorgianni, P. (1992). *J. Org. Chem.* **57**, 5128–5136.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Chen, J. J., Coles, P. J., Arnold, L. D., Smith, R. A., MacDonald, I. D., Carriére, J. & Krantz, A. (1996). *Bioorg. Med. Chem. Lett.* **6**, 435–438.
Concellón, J. M. & Bardales, E. (2003*a*). *Org. Lett.* **5**, 4783–4785.
Concellón, J. M. & Bardales, E. (2003*b*). *Tetrahedron Lett.* **44**, 5323–5326.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Martín, L. O., Chammaa, S., Pino, M. S. G., Sánchez, A. R., García, M. C., Assiego, C. & Sarabia, F. (2004). *Tetrahedron Lett.* **45**, 9069–9072.
Romero, N., Terán, J. L., Gnecco, D. & Bernès, S. (2005*a*). *Acta Cryst.* E**61**, o2924–o2926.
Romero, N., Terán, J. L., Gnecco, D. & Bernès, S. (2005*b*). *Acta Cryst.* E**61**, o2927–o2929.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheppard, G. S., Wang, J., Kawai, M., BaMaung, N. T., Craig, R. A., Erickson, S. A., Lynch, L., Patel, J., Yang, F., Searle, X. B., Lou, P., Park, C., Kim, K. H., Henkin, J. & Lesniewski, R. (2004). *Bioorg. Med. Chem. Lett.* **14**, 865–868.
Siemens (1994). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Szymanski, W. & Ostaszewski, R. (2006). *Tetrahedron Asymmetry*, **17**, 2667–2671.

# supporting information

*Acta Cryst.* (2009). E**65**, o743 [doi:10.1107/S1600536809008198]

## (1′*S*,2*R*,3*R*)-(−)-2-Hydroxy-3-morpholino-3-phenyl-*N*-(1′-phenylethyl)-propionamide

**Angel Mendoza, David M. Aparicio, Joel L. Terán, Dino Gnecco and Jorge R. Juárez**

### S1. Comment

The stereoselective synthesis of *α*-hydroxyamides (Barbaro *et al.*, 1992; Szymanski *et al.*, 2006) is an important area in asymmetric synthesis because these kinds of intermediates exhibit a great value as synthetic building block in the synthesis of pharmaceutical, agriculture and medicinal compounds (Sheppard *et al.*, 2004; Chen *et al.*, 1996). Numerous methods have been development through the last 10 years. One of the most relevant methodologies is the opening reaction of epoxyamides (Concellón *et al.*, 2003*a*,*b*; Martín *et al.*, 2004). In this way, we show herein the regiospecific ring opening reaction of epoxyamide derived from (*S*)-(-)-phenyl ethyl amine (Scheme 1). Related structures including (*S*)-(-)-phenyl ethyl amine with different substituents has been previously reported (Romero *et al.*, 2005*a*,*b*).

In the present paper, we report the structure of title compound, (I), which shows a single asymmetric unit with two aromatic rings. The morpholine ring shows an almost perfect chair conformation with a puckering parameters (Cremer & Pople, 1975) (O3—C2—C1—N2—C4—C3) $Q$ = 0.572 (4) Å, $\theta_2$ = 3.6° (4), $\varphi_2$ =343° (10), $q_2$ = 0.026 (4) Å and $q_3$ = 0.571 (4) Å. In the crystal structure, the molecules are linked by O—H···O hydrogen bonds (Table 1), generating a ribbon structure along the *a* axis.

### S2. Experimental

The compound (I) was obtained by *trans*-diastereoisomeric mixture of epoxyamide in anhydrous EtOH. Then, 1.1 equivalents of morpholine were added. The reaction was stirred over night. Finally, the resultant amino alcohol mixture was separate by column chromatography (AcOEt: Petroleum Ether). The absolute configuration of (1′*S*, 2*R*, 3*R*)-(-)-2-hydroxy-3-morpholin-4-yl-3 -phenyl-*N*-(1′-phenyl-ethyl)-propionamide was established by the structure determination of a (*S*)-(-)-phenyl ethyl amine of known absolute configuration of starting material [C8 in compound (I)]. The $H^1$ NMR experiment shows only one disteromeric compound.

### S3. Refinement

H atoms bonded to N and O atoms and all optically relevant were located in a difference map and refined as riding on their parent atoms with $U_{iso}$(H) = 1.2$U_{eq}$ for N and C—H and $U_{iso}$(H) = 1.5$U_{eq}$ for O atoms. H atoms linked to C atoms were placed in geometrical idealized positions and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å and with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C). In the absence of significant anomalous scattering effects, Friedel pairs were merged and the absolute configuration was assigned on the base of synthetic procedure.

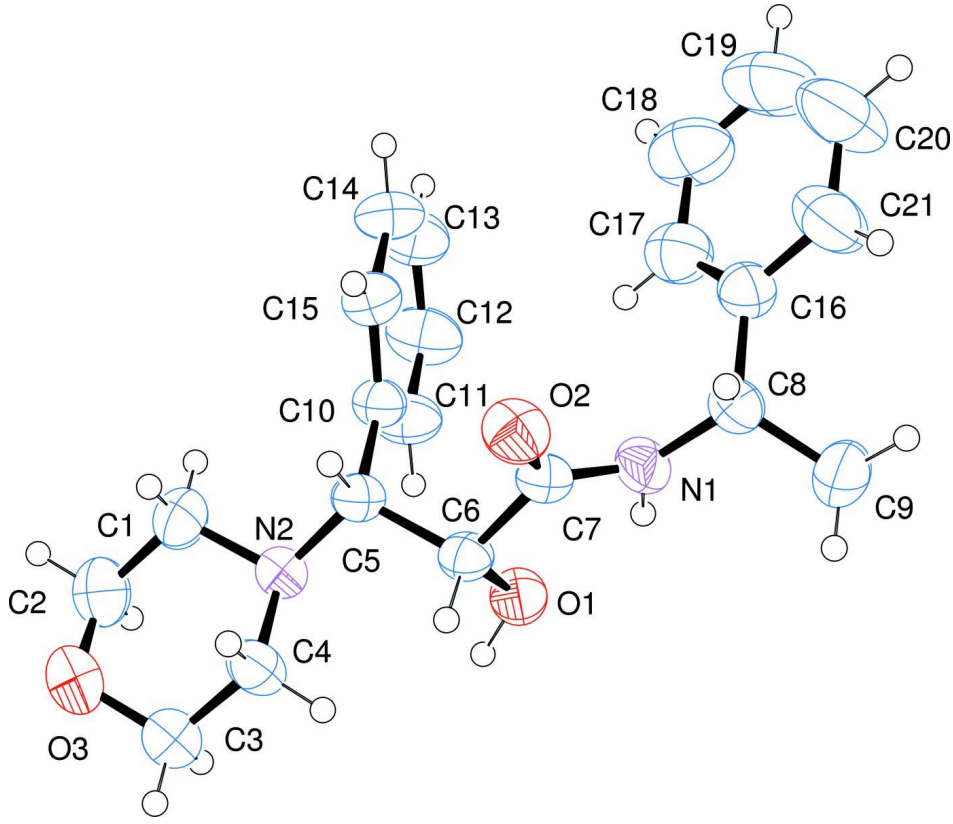



**Figure 1**

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

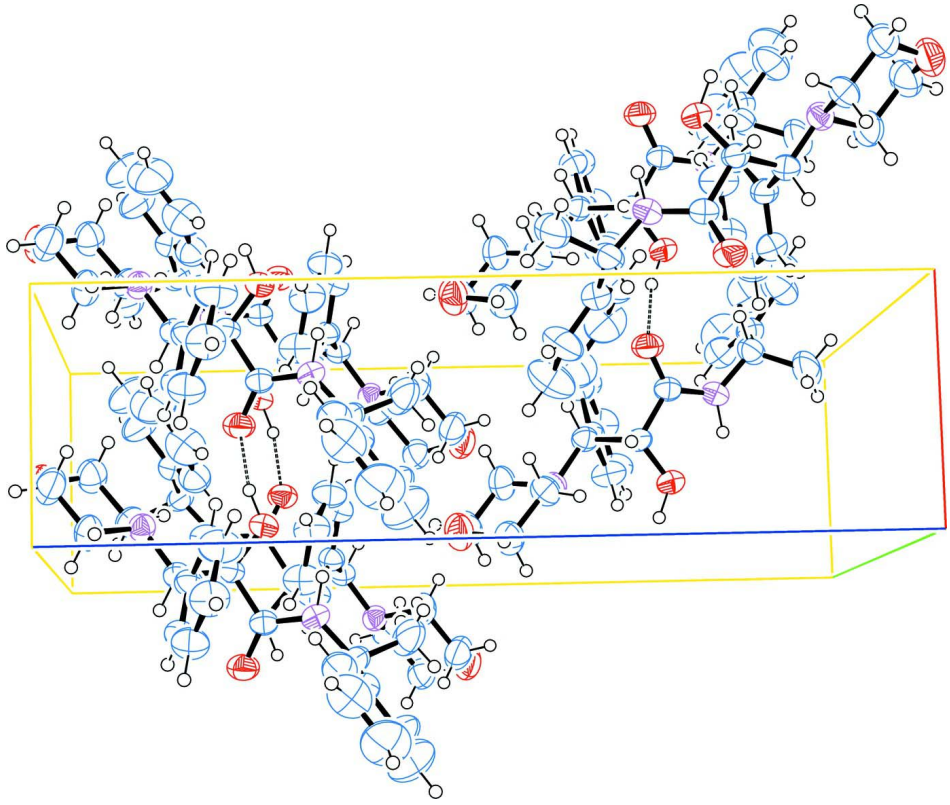


**Figure 2**

The packing of the title compound, showing molecules connected by O—H···O hydrogen bonds (dashed lines).

**(1′*S*,2*R*,3*R*)-(-)-2-Hydroxy-3-morpholino-3-phenyl- *N*-(1′-phenylethyl)propionamide**

*Crystal data*

$C_{21}H_{26}N_2O_3$
$M_r = 354.44$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.0010\ (17)$ Å
$b = 15.659\ (3)$ Å
$c = 20.746\ (4)$ Å
$V = 1949.5\ (8)$ Å$^3$
$Z = 4$
$F(000) = 760$
$D_x = 1.208$ Mg m$^{-3}$
Melting point: 431 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 3.9$–$23.9°$
$\mu = 0.08$ mm$^{-1}$
$T = 293$ K
Prism, colorless
0.72 × 0.28 × 0.16 mm

*Data collection*

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$2\theta/\omega$ scans
3964 measured reflections
2959 independent reflections
1116 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 29.0°$, $\theta_{min} = 1.6°$
$h = -1 \rightarrow 8$
$k = -1 \rightarrow 21$
$l = -1 \rightarrow 28$
3 standard reflections every 97 reflections
intensity decay: 3%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.163$
$S = 0.89$
2959 reflections
251 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
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.008 (2)

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 | −0.1563 (5) | 0.92034 (19) | 0.75127 (13) | 0.0567 (8) |
| N2 | −0.1559 (5) | 0.90373 (19) | 0.60796 (14) | 0.0464 (8) |
| O2 | 0.4127 (5) | 0.96834 (18) | 0.72379 (13) | 0.0598 (8) |
| N1 | 0.2201 (6) | 0.9177 (2) | 0.81024 (16) | 0.0516 (9) |
| C10 | 0.0914 (6) | 0.8018 (2) | 0.66365 (17) | 0.0447 (9) |
| C7 | 0.2354 (6) | 0.9439 (2) | 0.74924 (19) | 0.0445 (9) |
| O3 | −0.3564 (6) | 0.9556 (2) | 0.48787 (14) | 0.0770 (10) |
| C5 | 0.0430 (7) | 0.8947 (2) | 0.64872 (18) | 0.0449 (9) |
| C6 | 0.0197 (6) | 0.9474 (2) | 0.71116 (18) | 0.0434 (9) |
| C4 | −0.2007 (7) | 0.9933 (2) | 0.5920 (2) | 0.0554 (11) |
| H4A | −0.0697 | 1.0183 | 0.5721 | 0.066* |
| H4B | −0.2322 | 1.0248 | 0.6312 | 0.066* |
| C8 | 0.3993 (7) | 0.9216 (3) | 0.8565 (2) | 0.0552 (11) |
| C15 | 0.3024 (8) | 0.7691 (2) | 0.65361 (19) | 0.0535 (10) |
| H15 | 0.4145 | 0.8045 | 0.6380 | 0.064* |
| C16 | 0.5180 (7) | 0.8364 (3) | 0.86521 (19) | 0.0558 (11) |
| C11 | −0.0702 (7) | 0.7484 (3) | 0.6885 (2) | 0.0610 (11) |
| H11 | −0.2125 | 0.7695 | 0.6964 | 0.073* |
| C17 | 0.4526 (9) | 0.7626 (3) | 0.8349 (2) | 0.0703 (13) |
| H17 | 0.3292 | 0.7635 | 0.8078 | 0.084* |
| C3 | −0.3969 (8) | 1.0009 (3) | 0.5463 (2) | 0.0676 (13) |
| H3A | −0.5294 | 0.9782 | 0.5669 | 0.081* |
| H3B | −0.4235 | 1.0607 | 0.5367 | 0.081* |

| | | | | |
|---|---|---|---|---|
| C1 | −0.1284 (8) | 0.8570 (3) | 0.54712 (19) | 0.0637 (12) |
| H1A | −0.1097 | 0.7967 | 0.5564 | 0.076* |
| H1B | 0.0055 | 0.8768 | 0.5257 | 0.076* |
| C2 | −0.3229 (9) | 0.8683 (3) | 0.5030 (2) | 0.0765 (15) |
| H2A | −0.2973 | 0.8365 | 0.4635 | 0.092* |
| H2B | −0.4559 | 0.8457 | 0.5233 | 0.092* |
| C13 | 0.1855 (9) | 0.6310 (3) | 0.6898 (2) | 0.0699 (14) |
| H13 | 0.2158 | 0.5736 | 0.6973 | 0.084* |
| C14 | 0.3492 (8) | 0.6836 (3) | 0.6666 (2) | 0.0638 (12) |
| H14 | 0.4918 | 0.6623 | 0.6594 | 0.077* |
| C21 | 0.7048 (9) | 0.8339 (4) | 0.9047 (3) | 0.0896 (17) |
| H21 | 0.7533 | 0.8835 | 0.9250 | 0.107* |
| C9 | 0.3070 (9) | 0.9542 (4) | 0.9206 (2) | 0.0938 (18) |
| H9A | 0.2331 | 1.0078 | 0.9138 | 0.141* |
| H9B | 0.4271 | 0.9618 | 0.9506 | 0.141* |
| H9C | 0.2027 | 0.9135 | 0.9375 | 0.141* |
| C12 | −0.0219 (9) | 0.6627 (3) | 0.7018 (3) | 0.0742 (14) |
| H12 | −0.1316 | 0.6274 | 0.7189 | 0.089* |
| C18 | 0.5674 (12) | 0.6867 (3) | 0.8440 (3) | 0.0958 (18) |
| H18 | 0.5207 | 0.6370 | 0.8236 | 0.115* |
| C19 | 0.7512 (13) | 0.6857 (5) | 0.8837 (4) | 0.119 (2) |
| H19 | 0.8297 | 0.6351 | 0.8899 | 0.143* |
| C20 | 0.8184 (12) | 0.7586 (5) | 0.9141 (3) | 0.122 (2) |
| H20 | 0.9418 | 0.7574 | 0.9412 | 0.147* |
| H1O | −0.307 (14) | 0.929 (5) | 0.735 (4) | 0.184* |
| H1N | 0.059 (12) | 0.906 (4) | 0.819 (3) | 0.147* |
| H5 | 0.180 (12) | 0.915 (4) | 0.627 (3) | 0.147* |
| H6 | −0.004 (11) | 1.009 (4) | 0.697 (3) | 0.147* |
| H8 | 0.535 (12) | 0.960 (4) | 0.839 (3) | 0.147* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0370 (14) | 0.0800 (19) | 0.0531 (15) | 0.0034 (16) | 0.0045 (14) | 0.0008 (15) |
| N2 | 0.0476 (19) | 0.0465 (17) | 0.0450 (16) | 0.0034 (17) | −0.0023 (16) | 0.0024 (14) |
| O2 | 0.0429 (17) | 0.0697 (18) | 0.0668 (18) | −0.0070 (15) | 0.0011 (15) | 0.0061 (15) |
| N1 | 0.0388 (18) | 0.065 (2) | 0.0507 (19) | 0.0006 (18) | −0.0067 (17) | −0.0002 (17) |
| C10 | 0.041 (2) | 0.045 (2) | 0.049 (2) | 0.003 (2) | 0.0017 (19) | 0.0029 (17) |
| C7 | 0.038 (2) | 0.044 (2) | 0.051 (2) | 0.0032 (18) | −0.001 (2) | −0.0001 (19) |
| O3 | 0.091 (3) | 0.086 (2) | 0.0543 (17) | 0.013 (2) | −0.0085 (19) | 0.0103 (16) |
| C5 | 0.041 (2) | 0.046 (2) | 0.048 (2) | 0.0057 (19) | 0.0005 (19) | 0.0005 (17) |
| C6 | 0.034 (2) | 0.047 (2) | 0.049 (2) | 0.0020 (19) | 0.0026 (18) | 0.0012 (18) |
| C4 | 0.058 (3) | 0.050 (2) | 0.058 (2) | 0.007 (2) | −0.009 (2) | 0.008 (2) |
| C8 | 0.047 (2) | 0.064 (3) | 0.055 (2) | 0.008 (2) | −0.011 (2) | −0.009 (2) |
| C15 | 0.050 (2) | 0.052 (2) | 0.058 (2) | 0.002 (2) | 0.005 (2) | −0.001 (2) |
| C16 | 0.048 (3) | 0.067 (3) | 0.053 (2) | 0.005 (2) | 0.002 (2) | 0.003 (2) |
| C11 | 0.048 (3) | 0.050 (2) | 0.085 (3) | 0.002 (2) | 0.008 (3) | 0.009 (2) |
| C17 | 0.069 (3) | 0.068 (3) | 0.074 (3) | 0.007 (3) | 0.004 (3) | 0.008 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.073 (3) | 0.072 (3) | 0.058 (2) | 0.016 (3) | −0.003 (3) | 0.005 (2) |
| C1 | 0.080 (3) | 0.061 (2) | 0.051 (2) | 0.007 (3) | −0.003 (3) | −0.010 (2) |
| C2 | 0.083 (4) | 0.088 (3) | 0.058 (3) | 0.004 (3) | −0.016 (3) | −0.010 (3) |
| C13 | 0.072 (3) | 0.047 (2) | 0.090 (3) | 0.006 (3) | −0.002 (3) | 0.008 (2) |
| C14 | 0.057 (3) | 0.051 (2) | 0.084 (3) | 0.016 (2) | 0.004 (3) | −0.004 (2) |
| C21 | 0.075 (4) | 0.098 (4) | 0.096 (4) | 0.018 (4) | −0.030 (3) | 0.003 (3) |
| C9 | 0.080 (3) | 0.134 (5) | 0.068 (3) | 0.034 (4) | −0.018 (3) | −0.042 (3) |
| C12 | 0.063 (3) | 0.052 (3) | 0.107 (4) | −0.003 (2) | 0.003 (3) | 0.018 (3) |
| C18 | 0.108 (5) | 0.063 (3) | 0.116 (4) | 0.014 (4) | 0.016 (4) | 0.011 (3) |
| C19 | 0.114 (6) | 0.094 (5) | 0.149 (6) | 0.045 (5) | −0.001 (5) | 0.025 (5) |
| C20 | 0.104 (5) | 0.125 (5) | 0.137 (6) | 0.035 (5) | −0.042 (5) | 0.025 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C6 | 1.410 (5) | C16—C21 | 1.389 (6) |
| O1—H1O | 0.98 (8) | C11—C12 | 1.401 (6) |
| N2—C4 | 1.466 (5) | C11—H11 | 0.9300 |
| N2—C1 | 1.468 (5) | C17—C18 | 1.387 (7) |
| N2—C5 | 1.470 (5) | C17—H17 | 0.9300 |
| O2—C7 | 1.248 (4) | C3—H3A | 0.9700 |
| N1—C7 | 1.333 (5) | C3—H3B | 0.9700 |
| N1—C8 | 1.443 (5) | C1—C2 | 1.494 (7) |
| N1—H1N | 1.00 (7) | C1—H1A | 0.9700 |
| C10—C11 | 1.380 (5) | C1—H1B | 0.9700 |
| C10—C15 | 1.382 (6) | C2—H2A | 0.9700 |
| C10—C5 | 1.514 (5) | C2—H2B | 0.9700 |
| C7—C6 | 1.517 (5) | C13—C12 | 1.363 (7) |
| O3—C2 | 1.416 (5) | C13—C14 | 1.370 (6) |
| O3—C3 | 1.426 (5) | C13—H13 | 0.9300 |
| C5—C6 | 1.542 (5) | C14—H14 | 0.9300 |
| C5—H5 | 0.99 (7) | C21—C20 | 1.376 (8) |
| C6—H6 | 1.02 (6) | C21—H21 | 0.9300 |
| C4—C3 | 1.516 (6) | C9—H9A | 0.9600 |
| C4—H4A | 0.9700 | C9—H9B | 0.9600 |
| C4—H4B | 0.9700 | C9—H9C | 0.9600 |
| C8—C16 | 1.523 (6) | C12—H12 | 0.9300 |
| C8—C9 | 1.528 (6) | C18—C19 | 1.376 (9) |
| C8—H8 | 1.08 (7) | C18—H18 | 0.9300 |
| C15—C14 | 1.393 (5) | C19—C20 | 1.365 (9) |
| C15—H15 | 0.9300 | C19—H19 | 0.9300 |
| C16—C17 | 1.373 (6) | C20—H20 | 0.9300 |
| | | | |
| C6—O1—H1O | 116 (4) | C16—C17—H17 | 119.5 |
| C4—N2—C1 | 107.6 (3) | C18—C17—H17 | 119.5 |
| C4—N2—C5 | 111.8 (3) | O3—C3—C4 | 111.1 (4) |
| C1—N2—C5 | 110.8 (3) | O3—C3—H3A | 109.4 |
| C7—N1—C8 | 124.5 (4) | C4—C3—H3A | 109.4 |
| C7—N1—H1N | 107 (4) | O3—C3—H3B | 109.4 |

| | | | |
|---|---|---|---|
| C8—N1—H1N | 127 (4) | C4—C3—H3B | 109.4 |
| C11—C10—C15 | 118.4 (4) | H3A—C3—H3B | 108.0 |
| C11—C10—C5 | 121.5 (4) | N2—C1—C2 | 112.3 (4) |
| C15—C10—C5 | 120.1 (4) | N2—C1—H1A | 109.1 |
| O2—C7—N1 | 123.7 (4) | C2—C1—H1A | 109.1 |
| O2—C7—C6 | 119.7 (3) | N2—C1—H1B | 109.1 |
| N1—C7—C6 | 116.5 (3) | C2—C1—H1B | 109.1 |
| C2—O3—C3 | 108.5 (3) | H1A—C1—H1B | 107.9 |
| N2—C5—C10 | 111.5 (3) | O3—C2—C1 | 111.2 (4) |
| N2—C5—C6 | 111.0 (3) | O3—C2—H2A | 109.4 |
| C10—C5—C6 | 111.1 (3) | C1—C2—H2A | 109.4 |
| N2—C5—H5 | 112 (4) | O3—C2—H2B | 109.4 |
| C10—C5—H5 | 104 (4) | C1—C2—H2B | 109.4 |
| C6—C5—H5 | 107 (4) | H2A—C2—H2B | 108.0 |
| O1—C6—C7 | 108.7 (3) | C12—C13—C14 | 120.0 (4) |
| O1—C6—C5 | 113.8 (3) | C12—C13—H13 | 120.0 |
| C7—C6—C5 | 109.9 (3) | C14—C13—H13 | 120.0 |
| O1—C6—H6 | 110 (4) | C13—C14—C15 | 120.1 (4) |
| C7—C6—H6 | 108 (4) | C13—C14—H14 | 120.0 |
| C5—C6—H6 | 106 (4) | C15—C14—H14 | 120.0 |
| N2—C4—C3 | 111.0 (3) | C20—C21—C16 | 120.5 (6) |
| N2—C4—H4A | 109.4 | C20—C21—H21 | 119.8 |
| C3—C4—H4A | 109.4 | C16—C21—H21 | 119.8 |
| N2—C4—H4B | 109.4 | C8—C9—H9A | 109.5 |
| C3—C4—H4B | 109.4 | C8—C9—H9B | 109.5 |
| H4A—C4—H4B | 108.0 | H9A—C9—H9B | 109.5 |
| N1—C8—C16 | 113.0 (3) | C8—C9—H9C | 109.5 |
| N1—C8—C9 | 108.8 (3) | H9A—C9—H9C | 109.5 |
| C16—C8—C9 | 111.1 (4) | H9B—C9—H9C | 109.5 |
| N1—C8—H8 | 112 (3) | C13—C12—C11 | 120.1 (5) |
| C16—C8—H8 | 100 (3) | C13—C12—H12 | 119.9 |
| C9—C8—H8 | 112 (3) | C11—C12—H12 | 119.9 |
| C10—C15—C14 | 120.8 (4) | C19—C18—C17 | 119.4 (6) |
| C10—C15—H15 | 119.6 | C19—C18—H18 | 120.3 |
| C14—C15—H15 | 119.6 | C17—C18—H18 | 120.3 |
| C17—C16—C21 | 118.5 (5) | C20—C19—C18 | 120.2 (6) |
| C17—C16—C8 | 123.3 (4) | C20—C19—H19 | 119.9 |
| C21—C16—C8 | 118.1 (4) | C18—C19—H19 | 119.9 |
| C10—C11—C12 | 120.6 (4) | C19—C20—C21 | 120.4 (6) |
| C10—C11—H11 | 119.7 | C19—C20—H20 | 119.8 |
| C12—C11—H11 | 119.7 | C21—C20—H20 | 119.8 |
| C16—C17—C18 | 121.1 (5) | | |

| | | | |
|---|---|---|---|
| C8—N1—C7—O2 | 6.1 (6) | N1—C8—C16—C17 | −3.5 (6) |
| C8—N1—C7—C6 | −170.8 (3) | C9—C8—C16—C17 | 119.1 (5) |
| C4—N2—C5—C10 | −178.6 (3) | N1—C8—C16—C21 | 175.0 (4) |
| C1—N2—C5—C10 | −58.5 (4) | C9—C8—C16—C21 | −62.5 (5) |
| C4—N2—C5—C6 | 57.0 (4) | C15—C10—C11—C12 | −1.3 (6) |

| | | | |
|---|---|---|---|
| C1—N2—C5—C6 | 177.1 (3) | C5—C10—C11—C12 | 179.9 (4) |
| C11—C10—C5—N2 | −51.9 (5) | C21—C16—C17—C18 | 1.0 (7) |
| C15—C10—C5—N2 | 129.3 (4) | C8—C16—C17—C18 | 179.5 (4) |
| C11—C10—C5—C6 | 72.4 (5) | C2—O3—C3—C4 | 60.3 (5) |
| C15—C10—C5—C6 | −106.4 (4) | N2—C4—C3—O3 | −59.3 (5) |
| O2—C7—C6—O1 | −177.3 (3) | C4—N2—C1—C2 | −54.6 (5) |
| N1—C7—C6—O1 | −0.2 (4) | C5—N2—C1—C2 | −177.1 (4) |
| O2—C7—C6—C5 | 57.6 (5) | C3—O3—C2—C1 | −59.9 (5) |
| N1—C7—C6—C5 | −125.4 (3) | N2—C1—C2—O3 | 59.0 (5) |
| N2—C5—C6—O1 | 63.3 (4) | C12—C13—C14—C15 | −1.7 (7) |
| C10—C5—C6—O1 | −61.3 (4) | C10—C15—C14—C13 | −0.3 (7) |
| N2—C5—C6—C7 | −174.5 (3) | C17—C16—C21—C20 | −1.3 (8) |
| C10—C5—C6—C7 | 60.9 (4) | C8—C16—C21—C20 | −179.8 (6) |
| C1—N2—C4—C3 | 54.3 (5) | C14—C13—C12—C11 | 2.1 (8) |
| C5—N2—C4—C3 | 176.2 (3) | C10—C11—C12—C13 | −0.6 (7) |
| C7—N1—C8—C16 | −99.7 (5) | C16—C17—C18—C19 | −0.6 (8) |
| C7—N1—C8—C9 | 136.4 (4) | C17—C18—C19—C20 | 0.4 (10) |
| C11—C10—C15—C14 | 1.7 (6) | C18—C19—C20—C21 | −0.7 (11) |
| C5—C10—C15—C14 | −179.4 (4) | C16—C21—C20—C19 | 1.1 (10) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O1 | 1.00 (7) | 1.92 (7) | 2.569 (5) | 120 (5) |
| O1—H1*O*···O2$^{i}$ | 0.98 (8) | 1.80 (8) | 2.753 (4) | 163 (7) |

Symmetry code: (i) $x-1, y, z$.