Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Hydroxy-*N*′-(2-hydroxy-3-methoxy-5-nitrobenzylidene)-3-methylbenzohydrazide

You-Yue Han,* Yong-Hong Li and Qiu-Rong Zhao

Department of Chemistry and Life Science, Chuzhou University, Chuzhou, Anhui 239000, People's Republic of China
Correspondence e-mail: hanyouyue@126.com

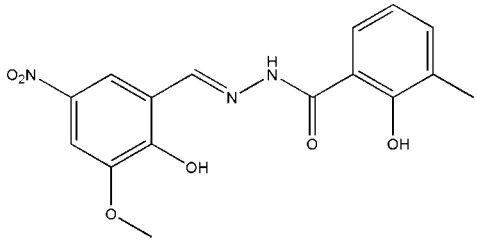
Received 6 April 2010; accepted 7 April 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.108; data-to-parameter ratio = 11.2.

In the title compound, $C_{16}H_{15}N_3O_6$, the dihedral angle between the two benzene rings is 0.9 (2)°. The molecule adopts an *E* configuration with respect to the C=N bond. There are intramolecular O—H···N and O—H···O hydrogen bonds in the molecule. In the crystal structure, molecules are linked through intermolecular N—H···O hydrogen bonds to form chains running along the *c* axis.

Related literature

For the biological properties of hydrazone compounds, see: Patil *et al.* (2010); Cukurovali *et al.* (2006). For the crystal structures of hydrazone compounds, see: Mohd Lair *et al.* (2009); Lin & Sang (2009); Suleiman Gwaram *et al.* (2010). For the hydrazone compounds we reported recently, see: Han & Zhao (2010*a*,*b*). For bond-length data, see: Allen *et al.* (1987). For similar compounds, see: Li & Ban (2009); Lo & Ng (2009); Ning & Xu (2009); Zhu *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{15}N_3O_6$
M_r = 345.31
Monoclinic, $P2_1/n$
a = 7.482 (1) Å
b = 17.158 (1) Å
c = 12.250 (1) Å
β = 91.565 (1)°
V = 1572.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.10 × 0.07 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.989, T_{max} = 0.994
14545 measured reflections
2612 independent reflections
2165 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.07
2612 reflections
233 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O6^{i}	0.90 (1)	2.22 (1)	3.0190 (17)	147 (2)
O4—H4···O3	0.82	1.79	2.5192 (15)	148
O1—H1···N2	0.82	1.90	2.6166 (17)	145

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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*.

This work was supported by the Applied Chemistry Key Subject of Anhui Province (grant No. 200802187C).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2671).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cukurovali, A., Yilmaz, I., Gur, S. & Kazaz, C. (2006). *Eur. J. Med. Chem.* **41**, 201–207.
Han, Y.-Y. & Zhao, Q.-R. (2010*a*). *Acta Cryst.* E**66**, o1025.
Han, Y.-Y. & Zhao, Q.-R. (2010*b*). *Acta Cryst.* E**66**, o1026.
Li, C.-M. & Ban, H.-Y. (2009). *Acta Cryst.* E**65**, o876.
Lin, X.-S. & Sang, Y.-L. (2009). *Acta Cryst.* E**65**, o1650.
Lo, K. M. & Ng, S. W. (2009). *Acta Cryst.* E**65**, o969.
Mohd Lair, N., Mohd Ali, H. & Ng, S. W. (2009). *Acta Cryst.* E**65**, o190.
Ning, J.-H. & Xu, X.-W. (2009). *Acta Cryst.* E**65**, o905–o906.
Patil, S. A., Naik, V. H., Kulkarni, A. D., Kamble, U., Bagihalli, G. B. & Badami, P. S. (2010). *J. Coord. Chem.* **63**, 688–699.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Suleiman Gwaram, N., Khaledi, H., Mohd Ali, H., Robinson, W. T. & Abdulla, M. A. (2010). *Acta Cryst.* E**66**, o721.
Zhu, C.-G., Wei, Y.-J. & Zhu, Q.-Y. (2009). *Acta Cryst.* E**65**, o85.

supporting information

Acta Cryst. (2010). E**66**, o1084 [https://doi.org/10.1107/S1600536810012912]

2-Hydroxy-*N*′-(2-hydroxy-3-methoxy-5-nitrobenzylidene)-3-methylbenzohydrazide

You-Yue Han, Yong-Hong Li and Qiu-Rong Zhao

S1. Comment

Hydrazone compounds have been widely investigated for their biological properties (Patil *et al.*, 2010; Cukurovali *et al.*, 2006). Furthermore, the crystal structures of the hydrazone compounds have also attracted much attention in recent years (Mohd Lair *et al.*, 2009; Lin & Sang, 2009; Suleiman Gwaram *et al.*, 2010). As a continuous work on the structural characterization of such compounds (Han & Zhao, 2010*a*,*b*), the title new hydrazone compound is reported.

In the title compound, Fig. 1, the dihedral angle between the two benzene rings is 0.9 (2)°. The molecule adopts an *E* configuration with respect to the C═N bond. There are intramolecular O—H···N and O—H···O hydrogen bonds in the molecule (Table 1). All the bond lengths are within normal ranges (Allen *et al.*, 1987), and are comparable with those in the similar compounds (Li & Ban, 2009; Lo & Ng, 2009; Ning & Xu, 2009; Zhu *et al.*, 2009).

In the crystal structure, molecules are linked through intermolecular N—H···O hydrogen bonds (Table 1) to form chains running along the *c* axis (Fig. 2).

S2. Experimental

A mixture of 3-methoxy-5-nitrosalicylaldehyde (0.197 g, 1 mmol) and 2-hydroxy-3-methylbenzohydrazide (0.166 g, 1 mmol) in 50 ml methanol was stirred at room temperature for 1 h. The mixture was filtered to remove impurities, and then left at room temperature. After a few days, single crystals of the title compound, suitable for X-ray diffraction, were formed.

S3. Refinement

Amino H atom was located from a difference Fourier map and refined isotropically, with N—H distance restrained to 0.90 (1) Å. Other H atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93 or 0.96 Å, O—H = 0.82 Å, and U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(methyl C and O).

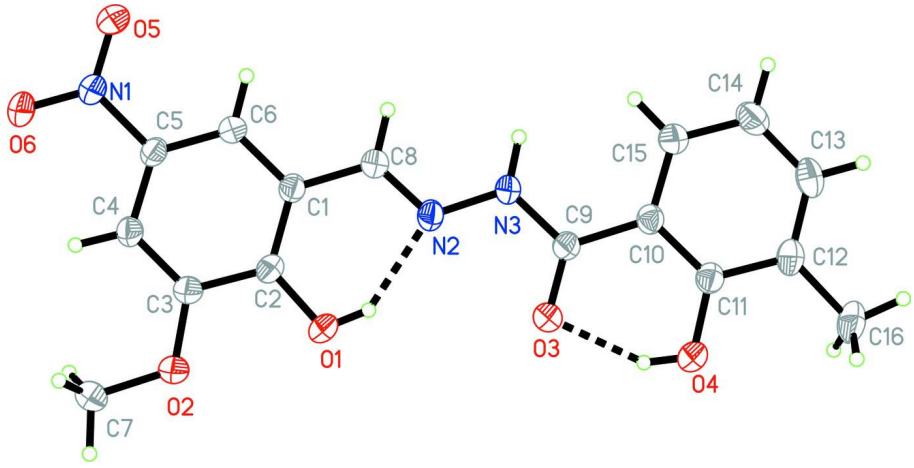


Figure 1

The molecular structure of the title compound with atom labels and the 30% probability displacement ellipsoids for non-H atoms. Intramolecular hydrogen bonds are shown as dashed lines.

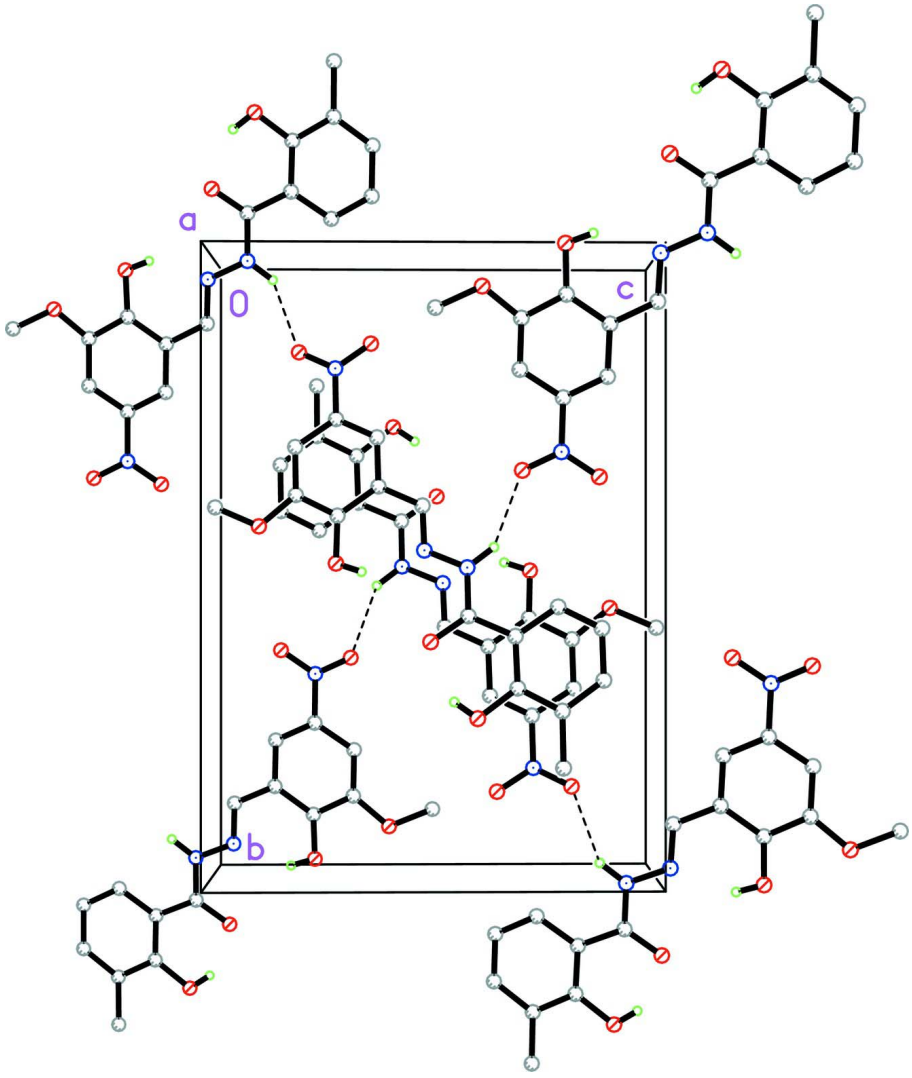


Figure 2

The molecular packing of the title compound. Hydrogen bonds are shown as dashed lines.

2-Hydroxy-*N*′-(2-hydroxy-3-methoxy-5-nitrobenzylidene)- 3-methylbenzohydrazide

Crystal data

$C_{16}H_{15}N_3O_6$
$M_r = 345.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.482$ (1) Å
$b = 17.158$ (1) Å
$c = 12.250$ (1) Å
$\beta = 91.565$ (1)°
$V = 1572.0$ (3) Å^3
$Z = 4$

$F(000) = 720$
$D_x = 1.459$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6384 reflections
$\theta = 2.4$–28.1°
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Block, colourless
0.10 × 0.07 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.989, T_{max} = 0.994
14545 measured reflections
2612 independent reflections
2165 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −8→8
k = −20→19
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.07
2612 reflections
233 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.2485P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.91330 (15)	0.99426 (5)	0.21838 (9)	0.0529 (3)
H1	0.8639	1.0075	0.1607	0.079*
O2	1.05158 (16)	0.93423 (6)	0.39521 (9)	0.0569 (3)
O3	0.67111 (17)	1.11702 (6)	0.00680 (9)	0.0596 (3)
O4	0.55183 (17)	1.23435 (6)	−0.09508 (10)	0.0633 (3)
H4	0.5986	1.2096	−0.0447	0.095*
O5	1.0371 (2)	0.63937 (6)	0.14205 (11)	0.0805 (4)
O6	1.13335 (17)	0.65067 (6)	0.30689 (10)	0.0654 (4)
N1	1.06881 (16)	0.67848 (7)	0.22311 (11)	0.0481 (3)
N2	0.78019 (15)	0.97366 (7)	0.02075 (10)	0.0428 (3)
N3	0.70013 (16)	1.00078 (6)	−0.07337 (10)	0.0434 (3)
C1	0.91165 (17)	0.87130 (8)	0.12358 (11)	0.0391 (3)
C2	0.94794 (18)	0.91768 (8)	0.21567 (12)	0.0397 (3)
C3	1.02423 (19)	0.88404 (8)	0.31098 (12)	0.0422 (3)
C4	1.06361 (18)	0.80575 (8)	0.31361 (12)	0.0425 (4)

H4A	1.1130	0.7830	0.3764	0.051*
C5	1.02806 (18)	0.76137 (8)	0.22055 (12)	0.0406 (3)
C6	0.95409 (18)	0.79211 (8)	0.12723 (12)	0.0423 (3)
H6	0.9321	0.7606	0.0665	0.051*
C7	1.1154 (3)	0.90321 (10)	0.49667 (14)	0.0671 (5)
H7A	1.0319	0.8654	0.5224	0.101*
H7B	1.1281	0.9445	0.5491	0.101*
H7C	1.2293	0.8788	0.4870	0.101*
C8	0.8290 (2)	0.90257 (8)	0.02500 (12)	0.0444 (4)
H8	0.8113	0.8706	−0.0357	0.053*
C9	0.64676 (18)	1.07636 (8)	−0.07544 (12)	0.0398 (3)
C10	0.56400 (18)	1.10707 (8)	−0.17680 (12)	0.0393 (3)
C11	0.52198 (18)	1.18678 (8)	−0.18113 (12)	0.0433 (4)
C12	0.44698 (19)	1.22062 (9)	−0.27568 (13)	0.0486 (4)
C13	0.4115 (2)	1.17278 (10)	−0.36333 (14)	0.0566 (4)
H13	0.3604	1.1941	−0.4266	0.068*
C14	0.4493 (2)	1.09387 (10)	−0.36069 (14)	0.0587 (4)
H14	0.4230	1.0629	−0.4214	0.070*
C15	0.5255 (2)	1.06140 (9)	−0.26864 (13)	0.0493 (4)
H15	0.5519	1.0084	−0.2673	0.059*
C16	0.4079 (2)	1.30676 (10)	−0.27722 (16)	0.0659 (5)
H16A	0.3563	1.3210	−0.3470	0.099*
H16B	0.5169	1.3352	−0.2646	0.099*
H16C	0.3255	1.3190	−0.2210	0.099*
H3	0.686 (3)	0.9691 (10)	−0.1318 (12)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0724 (7)	0.0306 (5)	0.0552 (7)	0.0048 (4)	−0.0085 (5)	−0.0008 (4)
O2	0.0815 (8)	0.0413 (6)	0.0468 (7)	0.0050 (5)	−0.0152 (5)	−0.0065 (5)
O3	0.0876 (8)	0.0438 (6)	0.0464 (7)	0.0091 (5)	−0.0182 (6)	−0.0042 (5)
O4	0.0868 (8)	0.0393 (6)	0.0628 (8)	0.0129 (5)	−0.0201 (6)	−0.0052 (5)
O5	0.1370 (12)	0.0401 (6)	0.0630 (9)	0.0209 (7)	−0.0251 (8)	−0.0099 (6)
O6	0.0919 (9)	0.0429 (6)	0.0603 (8)	0.0125 (6)	−0.0219 (6)	0.0094 (5)
N1	0.0588 (8)	0.0361 (6)	0.0490 (9)	0.0068 (5)	−0.0062 (6)	0.0023 (6)
N2	0.0485 (7)	0.0384 (6)	0.0413 (8)	0.0007 (5)	−0.0027 (5)	0.0079 (5)
N3	0.0558 (7)	0.0357 (6)	0.0382 (8)	0.0026 (5)	−0.0060 (6)	0.0050 (5)
C1	0.0416 (7)	0.0353 (7)	0.0401 (9)	−0.0001 (5)	−0.0008 (6)	0.0039 (6)
C2	0.0417 (7)	0.0320 (7)	0.0455 (9)	0.0005 (5)	0.0009 (6)	0.0020 (6)
C3	0.0455 (8)	0.0390 (7)	0.0417 (9)	−0.0014 (6)	−0.0026 (6)	−0.0032 (6)
C4	0.0452 (8)	0.0398 (7)	0.0419 (9)	0.0021 (6)	−0.0059 (6)	0.0043 (6)
C5	0.0443 (7)	0.0320 (7)	0.0453 (9)	0.0034 (6)	−0.0022 (6)	0.0025 (6)
C6	0.0510 (8)	0.0359 (7)	0.0397 (9)	0.0019 (6)	−0.0029 (6)	−0.0019 (6)
C7	0.0994 (14)	0.0544 (10)	0.0465 (11)	0.0060 (9)	−0.0192 (9)	−0.0069 (8)
C8	0.0537 (9)	0.0385 (7)	0.0406 (9)	0.0015 (6)	−0.0034 (7)	0.0017 (6)
C9	0.0436 (8)	0.0359 (7)	0.0396 (9)	−0.0017 (6)	−0.0019 (6)	0.0019 (6)
C10	0.0418 (7)	0.0381 (7)	0.0381 (9)	−0.0014 (6)	0.0000 (6)	0.0051 (6)

C11	0.0434 (7)	0.0409 (7)	0.0454 (9)	0.0004 (6)	−0.0010 (6)	0.0033 (7)
C12	0.0448 (8)	0.0495 (8)	0.0514 (10)	0.0019 (6)	0.0005 (7)	0.0149 (7)
C13	0.0556 (9)	0.0691 (11)	0.0449 (10)	0.0022 (8)	−0.0037 (7)	0.0193 (8)
C14	0.0693 (11)	0.0656 (11)	0.0407 (10)	−0.0021 (8)	−0.0083 (8)	−0.0010 (8)
C15	0.0588 (9)	0.0442 (8)	0.0447 (10)	0.0004 (7)	−0.0039 (7)	0.0010 (7)
C16	0.0663 (11)	0.0524 (10)	0.0786 (13)	0.0071 (8)	−0.0083 (9)	0.0239 (9)

Geometric parameters (Å, º)

O1—C2	1.3399 (16)	C5—C6	1.363 (2)
O1—H1	0.8200	C6—H6	0.9300
O2—C3	1.3553 (17)	C7—H7A	0.9600
O2—C7	1.423 (2)	C7—H7B	0.9600
O3—C9	1.2348 (17)	C7—H7C	0.9600
O4—C11	1.3469 (18)	C8—H8	0.9300
O4—H4	0.8200	C9—C10	1.470 (2)
O5—N1	1.2165 (16)	C10—C15	1.395 (2)
O6—N1	1.2195 (16)	C10—C11	1.4040 (19)
N1—C5	1.4547 (18)	C11—C12	1.399 (2)
N2—C8	1.2739 (18)	C12—C13	1.372 (2)
N2—N3	1.3664 (17)	C12—C16	1.507 (2)
N3—C9	1.3569 (18)	C13—C14	1.383 (2)
N3—H3	0.903 (9)	C13—H13	0.9300
C1—C6	1.3958 (18)	C14—C15	1.368 (2)
C1—C2	1.401 (2)	C14—H14	0.9300
C1—C8	1.445 (2)	C15—H15	0.9300
C2—C3	1.409 (2)	C16—H16A	0.9600
C3—C4	1.3754 (19)	C16—H16B	0.9600
C4—C5	1.391 (2)	C16—H16C	0.9600
C4—H4A	0.9300		

C2—O1—H1	109.5	H7A—C7—H7C	109.5
C3—O2—C7	117.87 (12)	H7B—C7—H7C	109.5
C11—O4—H4	109.5	N2—C8—C1	120.36 (14)
O5—N1—O6	122.35 (12)	N2—C8—H8	119.8
O5—N1—C5	119.05 (12)	C1—C8—H8	119.8
O6—N1—C5	118.60 (13)	O3—C9—N3	119.19 (13)
C8—N2—N3	118.63 (13)	O3—C9—C10	122.42 (12)
C9—N3—N2	117.60 (12)	N3—C9—C10	118.38 (13)
C9—N3—H3	122.2 (13)	C15—C10—C11	118.41 (13)
N2—N3—H3	120.2 (13)	C15—C10—C9	123.57 (12)
C6—C1—C2	119.23 (13)	C11—C10—C9	118.02 (13)
C6—C1—C8	118.69 (13)	O4—C11—C12	116.78 (13)
C2—C1—C8	122.08 (12)	O4—C11—C10	121.90 (13)
O1—C2—C1	122.93 (13)	C12—C11—C10	121.31 (14)
O1—C2—C3	117.09 (13)	C13—C12—C11	117.67 (14)
C1—C2—C3	119.98 (12)	C13—C12—C16	122.95 (15)
O2—C3—C4	125.10 (14)	C11—C12—C16	119.39 (15)

O2—C3—C2	114.82 (12)	C12—C13—C14	122.16 (15)
C4—C3—C2	120.07 (13)	C12—C13—H13	118.9
C3—C4—C5	118.66 (13)	C14—C13—H13	118.9
C3—C4—H4A	120.7	C15—C14—C13	119.88 (16)
C5—C4—H4A	120.7	C15—C14—H14	120.1
C6—C5—C4	122.69 (12)	C13—C14—H14	120.1
C6—C5—N1	118.46 (13)	C14—C15—C10	120.55 (14)
C4—C5—N1	118.84 (13)	C14—C15—H15	119.7
C5—C6—C1	119.37 (13)	C10—C15—H15	119.7
C5—C6—H6	120.3	C12—C16—H16A	109.5
C1—C6—H6	120.3	C12—C16—H16B	109.5
O2—C7—H7A	109.5	H16A—C16—H16B	109.5
O2—C7—H7B	109.5	C12—C16—H16C	109.5
H7A—C7—H7B	109.5	H16A—C16—H16C	109.5
O2—C7—H7C	109.5	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O6^{i}	0.90 (1)	2.22 (1)	3.0190 (17)	147 (2)
O4—H4···O3	0.82	1.79	2.5192 (15)	148
O1—H1···N2	0.82	1.90	2.6166 (17)	145

Symmetry code: (i) $x-1/2, -y+3/2, z-1/2$.