Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-{[2-(2-Hydroxy-3-methoxybenzylidene)hydrazin-1-ylidene]methyl}-6-methoxyphenol

Rong Lu,[a]* Wenxia Wang,[a] Xingqiang Lü[a] and Shunsheng Zhao[b]

[a]College of Chemical Engineering, Northwest University, Xi'an 710069, Shaanxi, People's Republic of China, and [b]College of Chemistry and Chemical Engineering, Xian University of Science and Technology, Xi'an 710054, Shaanxi, People's Republic of China
Correspondence e-mail: lu78441@yahoo.com.cn

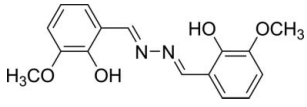
Received 17 July 2011; accepted 10 September 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.061; wR factor = 0.186; data-to-parameter ratio = 12.7.

The title compound, $C_{16}H_{16}N_2O_4$, was obtained from the reaction of hydrazine hydrate and *o*-vanilin in absolute ethanol. The molecule is almost planar (except for the methyl H atoms), with a mean deviation from the plane of 0.0259 Å. The molecular structure also exhibits an approximate non-crystallographic twofold axis. Intramolecular O—H···N hydrogen bonds occur. In the crystal, intermolecular C—H···O hydrogen bonds generate molecular zigzag sheets. The sheets stack through C—H···π interactions, leading to a three-dimensional-network.

Related literature

For the properties and applications of the title compound or similar structural compounds and their metal complexes, see: Lin *et al.* (2009); Davidson *et al.* (2006); Lin & Zeng (2006).



Experimental

Crystal data

$C_{16}H_{16}N_2O_4$
M_r = 300.31
Monoclinic, $P2_1/c$
a = 6.3095 (14) Å
b = 17.405 (4) Å
c = 13.606 (3) Å
β = 95.590 (4)°
V = 1487.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.858, T_{max} = 1.000
7393 measured reflections
2648 independent reflections
1133 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.186
S = 1.07
2648 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the C2–C7 and C10–C15 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*A*···N2	0.91 (5)	1.82 (5)	2.640 (4)	149 (4)
O2—H2*A*···N1	0.88 (4)	1.82 (4)	2.636 (4)	153 (4)
C16—H16*A*···O4^{i}	0.96	2.55	3.279 (5)	133
C7—H7*A*···*Cg*2^{ii}	0.93	2.90	3.694 (4)	144
C13—H13*A*···*Cg*1^{iii}	0.93	2.89	3.717 (4)	149

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This project was supported by the Natural Science Basic Research Plan in Shaanxi Province of China (program No. 2010JM2006, 2011JQ2011) and the Scientific Research Program funded by Shaanxi Provincial Education Department (Program No. 2008 J K440).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2353).

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Davidson, M. G., Johnson, A. L., Jones, M. D., Lunn, M. D. & Mahon, M. F. (2006). *Eur. J. Inorg. Chem.* **21**, 4449–4454.
Lin, P.-H., Burchell, T. J., Ungur, L., Chibotaru, L. F., Wernsdorfer, W. & Murugesu, M. (2009). *Angew. Chem. Int. Ed.* **48**, 9489–9452.
Lin, Z.-D. & Zeng, W. (2006). *Acta Cryst.* E**62**, m1074–m1076.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o2702 [https://doi.org/10.1107/S1600536811036816]

2-{[2-(2-Hydroxy-3-methoxybenzylidene)hydrazin-1-ylidene]methyl}-6-methoxyphenol

Rong Lu, Wenxia Wang, Xingqiang Lü and Shunsheng Zhao

S1. Comment

The title compound, (I) (Fig. 1), with various chelating atoms, could coordinate with many transition metals (Davidson *et al., 2006*) and lanthanides (Lin and Zeng, 2006; Lin *et al. , 2009*) to form functional complexes. The molecule crystallizes in the monoclinic space group P21/c and appears to be almost completely planar (except for the methyl hydrogen atoms) with a mean deviation from the plane ooff 0.0259 Å. The molecule also exhibits a non-crystallographic 2-fold axis. There are intramolecular O—H···N hydrogen bonds, intermolecular C—H···O hydrogen bonds and C—H···π hydrogen bonds. Molecules are linked by the C—H···O hydrogen bonds, generating molecular zigzag sheets, as shown in Fig. 2. The C—H···π hydrogen bonds and stacking interaction of these sheets leads to a three-dimensional-network. (Fig. 3).

S2. Experimental

The title compound was obtained from the reaction of hydrazine hydrate and *o*-vanilin in absolute ethanol. Hydrazine hydrate (500 mg, 10 mmol) was added to a solution of *o*-vanilin (3.04 g, 20 mmol) in absolute ethanol (200 ml) and heated to reflux for 2 h. The resulting solution was allowed to evaporate at rt to give a yellow crystal, which was collected by filtration and dried under vacumn;; yield 89.3%. The single-crystal of the title compound suitble for X-ray diffraction was obtained by recrystalization from absolute ethanol.

S3. Refinement

H atoms bonded to O atoms were refined isotropically without restraints, and with U_{iso}(H) = 1.2U_{eq}(O). Other H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

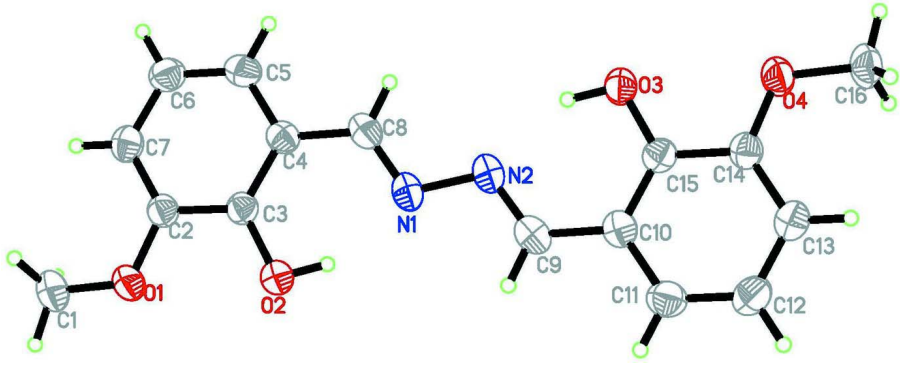


Figure 1

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

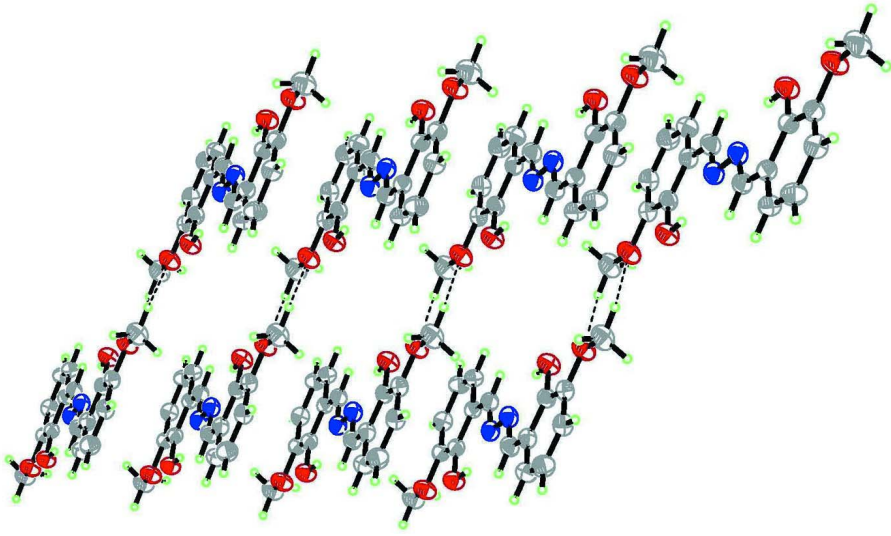


Figure 2

The packing of (I), showing one chain of molecules connected by C—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

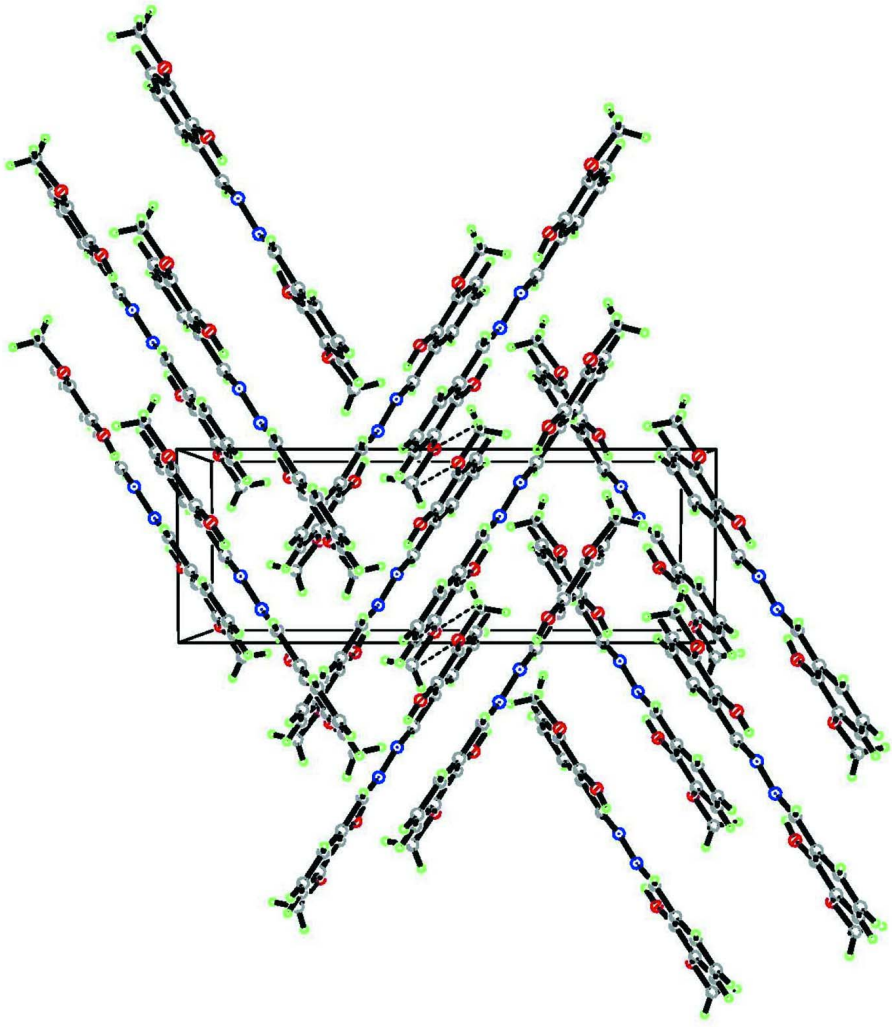


Figure 3

The packing of (I), showing one layer of molecules connected by stacking interaction.

2-{[2-(2-Hydroxy-3-methoxybenzylidene)hydrazin-1-ylidene]methyl}-6-methoxyphenol

Crystal data

$C_{16}H_{16}N_2O_4$
$M_r = 300.31$
Monoclinic, $P2_1/c$
$a = 6.3095\ (14)$ Å
$b = 17.405\ (4)$ Å
$c = 13.606\ (3)$ Å
$\beta = 95.590\ (4)°$
$V = 1487.0\ (6)$ Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.341$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8451 reflections
$\theta = 1.9–26.6°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, yellow
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
thin–slice ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.858, T_{max} = 1.000
7393 measured reflections
2648 independent reflections
1133 reflections with $I > 2\sigma(I)$

R_{int} = 0.052
θ_{max} = 25.1°, θ_{min} = 1.9°
h = −6→7
k = −18→20
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.186
S = 1.07
2648 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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}
O2	1.1229 (5)	0.30718 (16)	0.11097 (19)	0.0657 (8)
O3	0.3751 (4)	0.44682 (15)	0.36771 (19)	0.0659 (8)
N2	0.6605 (5)	0.39741 (18)	0.2529 (2)	0.0603 (9)
N1	0.8399 (5)	0.35841 (17)	0.2254 (2)	0.0593 (9)
C4	1.1395 (6)	0.28216 (19)	0.2861 (3)	0.0529 (10)
O1	1.4697 (4)	0.23030 (15)	0.09342 (18)	0.0708 (8)
C2	1.4056 (6)	0.2325 (2)	0.1861 (3)	0.0533 (10)
O4	0.0317 (4)	0.52385 (14)	0.38754 (18)	0.0696 (8)
C14	0.0966 (6)	0.5236 (2)	0.2954 (3)	0.0555 (10)
C3	1.2189 (6)	0.27497 (19)	0.1943 (3)	0.0513 (9)
C15	0.2831 (6)	0.4809 (2)	0.2850 (3)	0.0532 (10)
C13	−0.0034 (6)	0.5612 (2)	0.2144 (3)	0.0628 (11)
H13A	−0.1247	0.5904	0.2210	0.075*
C10	0.3625 (6)	0.4756 (2)	0.1936 (3)	0.0570 (10)
C8	0.9494 (6)	0.3256 (2)	0.2985 (3)	0.0585 (11)
H8A	0.9043	0.3300	0.3613	0.070*
C11	0.2542 (7)	0.5129 (2)	0.1132 (3)	0.0702 (12)
H11A	0.3043	0.5086	0.0514	0.084*

C5	1.2488 (6)	0.2463 (2)	0.3685 (3)	0.0638 (11)
H5A	1.1981	0.2512	0.4301	0.077*
C7	1.5070 (6)	0.1973 (2)	0.2683 (3)	0.0624 (11)
H7A	1.6292	0.1684	0.2627	0.075*
C9	0.5516 (6)	0.4314 (2)	0.1809 (3)	0.0633 (11)
H9A	0.5963	0.4273	0.1179	0.076*
C6	1.4279 (7)	0.2047 (2)	0.3592 (3)	0.0674 (11)
H6A	1.4980	0.1810	0.4144	0.081*
C16	−0.1522 (6)	0.5682 (2)	0.4042 (3)	0.0783 (14)
H16A	−0.1799	0.5635	0.4721	0.117*
H16B	−0.2728	0.5497	0.3624	0.117*
H16C	−0.1274	0.6211	0.3893	0.117*
C12	0.0759 (7)	0.5556 (2)	0.1232 (3)	0.0731 (12)
H12A	0.0079	0.5810	0.0688	0.088*
C1	1.6443 (6)	0.1816 (2)	0.0767 (3)	0.0817 (14)
H1B	1.6730	0.1854	0.0089	0.123*
H1C	1.6095	0.1294	0.0914	0.123*
H1D	1.7680	0.1972	0.1186	0.123*
H3A	0.490 (8)	0.421 (3)	0.349 (4)	0.128 (19)*
H2A	1.007 (7)	0.328 (2)	0.131 (3)	0.096 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0594 (19)	0.080 (2)	0.0581 (18)	0.0130 (15)	0.0097 (15)	0.0022 (14)
O3	0.0600 (19)	0.0752 (19)	0.0638 (19)	0.0177 (15)	0.0130 (15)	0.0115 (14)
N2	0.052 (2)	0.060 (2)	0.071 (2)	0.0020 (16)	0.0164 (17)	−0.0059 (17)
N1	0.049 (2)	0.060 (2)	0.071 (2)	0.0004 (16)	0.0146 (18)	−0.0089 (17)
C4	0.055 (2)	0.051 (2)	0.053 (2)	−0.0052 (18)	0.0082 (19)	−0.0062 (18)
O1	0.0661 (18)	0.0868 (19)	0.0619 (18)	0.0217 (15)	0.0187 (14)	0.0032 (14)
C2	0.050 (2)	0.055 (2)	0.055 (2)	0.0014 (19)	0.0069 (19)	−0.0041 (18)
O4	0.0630 (18)	0.0796 (19)	0.0691 (18)	0.0180 (15)	0.0214 (14)	0.0114 (14)
C14	0.049 (2)	0.055 (2)	0.063 (3)	−0.0023 (19)	0.008 (2)	0.0028 (19)
C3	0.050 (2)	0.052 (2)	0.052 (2)	−0.0001 (19)	0.0042 (18)	−0.0021 (18)
C15	0.052 (2)	0.051 (2)	0.056 (2)	−0.0026 (19)	0.0052 (19)	0.0064 (18)
C13	0.056 (3)	0.059 (3)	0.073 (3)	0.0026 (19)	0.003 (2)	0.007 (2)
C10	0.054 (2)	0.054 (2)	0.064 (3)	−0.0039 (19)	0.009 (2)	−0.0012 (19)
C8	0.056 (3)	0.059 (2)	0.062 (3)	−0.006 (2)	0.015 (2)	−0.011 (2)
C11	0.071 (3)	0.081 (3)	0.059 (3)	−0.003 (2)	0.010 (2)	0.002 (2)
C5	0.071 (3)	0.068 (3)	0.053 (3)	−0.007 (2)	0.008 (2)	−0.007 (2)
C7	0.058 (3)	0.059 (3)	0.070 (3)	0.008 (2)	0.005 (2)	0.000 (2)
C9	0.065 (3)	0.060 (3)	0.067 (3)	−0.005 (2)	0.020 (2)	−0.008 (2)
C6	0.074 (3)	0.068 (3)	0.059 (3)	0.010 (2)	0.001 (2)	0.0008 (19)
C16	0.054 (3)	0.093 (3)	0.091 (3)	0.018 (2)	0.021 (2)	0.006 (2)
C12	0.067 (3)	0.079 (3)	0.071 (3)	0.005 (2)	−0.005 (2)	0.009 (2)
C1	0.065 (3)	0.098 (3)	0.085 (3)	0.019 (3)	0.023 (2)	−0.004 (2)

Geometric parameters (Å, º)

O2—C3	1.354 (4)	C13—H13A	0.9300
O2—H2A	0.88 (4)	C10—C11	1.392 (5)
O3—C15	1.352 (4)	C10—C9	1.445 (5)
O3—H3A	0.91 (5)	C8—H8A	0.9300
N2—C9	1.285 (4)	C11—C12	1.366 (5)
N2—N1	1.402 (4)	C11—H11A	0.9300
N1—C8	1.288 (4)	C5—C6	1.359 (5)
C4—C3	1.396 (4)	C5—H5A	0.9300
C4—C5	1.404 (5)	C7—C6	1.384 (5)
C4—C8	1.441 (5)	C7—H7A	0.9300
O1—C2	1.361 (4)	C9—H9A	0.9300
O1—C1	1.426 (4)	C6—H6A	0.9300
C2—C7	1.377 (5)	C16—H16A	0.9600
C2—C3	1.404 (5)	C16—H16B	0.9600
O4—C14	1.357 (4)	C16—H16C	0.9600
O4—C16	1.430 (4)	C12—H12A	0.9300
C14—C13	1.380 (5)	C1—H1B	0.9600
C14—C15	1.411 (5)	C1—H1C	0.9600
C15—C10	1.388 (5)	C1—H1D	0.9600
C13—C12	1.385 (5)		
C3—O2—H2A	103 (3)	C12—C11—C10	121.4 (4)
C15—O3—H3A	106 (3)	C12—C11—H11A	119.3
C9—N2—N1	113.9 (3)	C10—C11—H11A	119.3
C8—N1—N2	113.3 (3)	C6—C5—C4	120.7 (3)
C3—C4—C5	118.9 (3)	C6—C5—H5A	119.6
C3—C4—C8	121.8 (4)	C4—C5—H5A	119.6
C5—C4—C8	119.3 (3)	C2—C7—C6	120.3 (4)
C2—O1—C1	117.9 (3)	C2—C7—H7A	119.8
O1—C2—C7	125.7 (3)	C6—C7—H7A	119.8
O1—C2—C3	114.6 (3)	N2—C9—C10	122.7 (3)
C7—C2—C3	119.8 (3)	N2—C9—H9A	118.6
C14—O4—C16	118.1 (3)	C10—C9—H9A	118.6
O4—C14—C13	125.5 (3)	C5—C6—C7	120.6 (4)
O4—C14—C15	115.1 (3)	C5—C6—H6A	119.7
C13—C14—C15	119.4 (3)	C7—C6—H6A	119.7
O2—C3—C4	122.8 (3)	O4—C16—H16A	109.5
O2—C3—C2	117.5 (3)	O4—C16—H16B	109.5
C4—C3—C2	119.7 (4)	H16A—C16—H16B	109.5
O3—C15—C10	123.6 (3)	O4—C16—H16C	109.5
O3—C15—C14	116.3 (3)	H16A—C16—H16C	109.5
C10—C15—C14	120.1 (4)	H16B—C16—H16C	109.5
C14—C13—C12	120.3 (4)	C11—C12—C13	120.0 (4)
C14—C13—H13A	119.8	C11—C12—H12A	120.0
C12—C13—H13A	119.8	C13—C12—H12A	120.0
C15—C10—C11	118.7 (4)	O1—C1—H1B	109.5

C15—C10—C9	121.1 (4)	O1—C1—H1C	109.5
C11—C10—C9	120.1 (3)	H1B—C1—H1C	109.5
N1—C8—C4	122.2 (3)	O1—C1—H1D	109.5
N1—C8—H8A	118.9	H1B—C1—H1D	109.5
C4—C8—H8A	118.9	H1C—C1—H1D	109.5

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C2–C7 and C10–C15 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···N2	0.91 (5)	1.82 (5)	2.640 (4)	149 (4)
O2—H2*A*···N1	0.88 (4)	1.82 (4)	2.636 (4)	153 (4)
C16—H16*A*···O4^{i}	0.96	2.55	3.279 (5)	133
C7—H7*A*···*Cg*2^{ii}	0.93	2.90	3.694 (4)	144
C13—H13*A*···*Cg*1^{iii}	0.93	2.89	3.717 (4)	149

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