



Crystal structure of *N*-(2-hydroxy-5-methylphenyl)benzamide

Rodolfo Moreno-Fuquen, Nory J. Mariño and Alan R. Kennedy

Acta Cryst. (2015). E71, o943



IUCr Journals

CRYSTALLOGRAPHY JOURNALS ONLINE

This open-access article is distributed under the terms of the Creative Commons Attribution Licence <http://creativecommons.org/licenses/by/2.0/uk/legalcode>, which permits unrestricted use, distribution, and reproduction in any medium, provided the original authors and source are cited.





Crystal structure of *N*-(2-hydroxy-5-methylphenyl)benzamide

Rodolfo Moreno-Fuquen,^{a*} Nory J. Mariño^a and Alan R. Kennedy^b

^aDepartamento de Química – Facultad de Ciencias Naturales y Exactas, Universidad del Valle, Apartado 25360, Santiago de Cali, Colombia, and ^bWestCHEM, Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland. *Correspondence e-mail: rodimo26@yahoo.es

Received 28 October 2015; accepted 29 October 2015

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

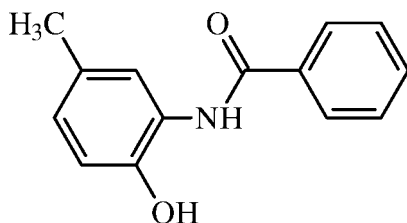
In the title compound, C₁₄H₁₃NO₂, the mean plane of the non-H atoms of the central amide fragment C–N–C(=O)–C (r.m.s. deviation = 0.029 Å) forms dihedral angles of 5.63 (6) and 10.20 (5)° with the phenyl and hydroxyphenyl rings, respectively. A short intramolecular N–H···O contact is present. In the crystal, the molecules are linked by O–H···O hydrogen bonds to generate *C*(7) chains along [100]. The chains are reinforced by weak C–H···O contacts, which together with the O–H···O bonds lead to *R*₂²(7) loops. Very weak N–H···O interactions link the molecules into inversion dimers.

Keywords: crystal structure; benzamide; benzanilide derivatives; biological activity.

CCDC reference: 1434264

1. Related literature

For the biological activity of benzanilide derivatives, see Calderone *et al.* (2006). For related structures, see: Gowda *et al.* (2008); Rodrigues *et al.* (2011).



2. Experimental

2.1. Crystal data

C₁₄H₁₃NO₂

M_r = 227.25

Monoclinic, *P*2₁/*n*
a = 7.2263 (3) Å
b = 21.7442 (7) Å
c = 7.4747 (3) Å
β = 110.280 (5)°
V = 1101.69 (8) Å³

Z = 4
Mo *Kα* radiation
μ = 0.09 mm^{−1}
T = 123 K
0.40 × 0.35 × 0.25 mm

2.2. Data collection

Oxford Diffraction Gemini S CCD diffractometer
10254 measured reflections

2795 independent reflections
2332 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.032

2.3. Refinement

R[*F*² > 2σ(*F*²)] = 0.043
wR(*F*²) = 0.109
S = 1.03
2795 reflections
163 parameters

H atoms treated by a mixture of independent and constrained refinement
*Δρ*_{max} = 0.33 e Å^{−3}
*Δρ*_{min} = −0.27 e Å^{−3}

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1···O2	0.889 (18)	2.173 (16)	2.6153 (15)	110.0 (13)
N1–H1···O2 ⁱ	0.889 (18)	2.518 (17)	3.1928 (14)	133.1 (14)
O2–H20···O1 ⁱⁱ	0.89 (2)	1.75 (2)	2.6390 (12)	171.3 (19)
C6–H6···O2 ⁱⁱⁱ	0.95	2.59	3.4197 (15)	146

Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $x + 1, y, z$; (iii) $x - 1, y, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

RMF is grateful to the Universidad del Valle, Colombia, for partial financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7532).

References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Calderone, V., Fiamingo, F. L., Giorgi, I., Leonardi, M., Livi, O., Martelli, A. & Martinotti, E. (2006). *Eur. J. Med. Chem.* **41**, 761–767.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Gowda, B. T., Foro, S., Sowmya, B. P. & Fuess, H. (2008). *Acta Cryst.* **E64**, o541.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Rodrigues, V. Z., Herich, P., Gowda, B. T. & Kožisek, J. (2011). *Acta Cryst.* **E67**, o3147.
Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.

supporting information

Acta Cryst. (2015). E71, o943 [doi:10.1107/S2056989015020575]

Crystal structure of *N*-(2-hydroxy-5-methylphenyl)benzamide

Rodolfo Moreno-Fuquen, Nory J. Mariño and Alan R. Kennedy

S1. Comment

The crystal structure determination of *N*-(2-hydroxy-5-methylphenyl)benzamide (I), is part of a study on phenylbenzamides carried out in our research group, and it was synthesized from the reaction between of 2-amino-4-methylphenol and benzoyl chloride in acetonitrile. Benzanilide systems have a wide range of biological properties such as potassium channel activators (Calderone *et al.*, 2006). Similar compounds to (I) have been reported in the literature: 2-Methyl-*N*-(*m*-tolyl)benzamide (II) (Gowda *et al.*, 2008) and *N*-(3,5-Dimethylphenyl)-4-methylbenzamide (III) (Rodrigues *et al.*, 2011). The molecular structure of (I) is shown in Fig. 1. The central amide moiety, C8—N1-C7(=O1)—C1, is close to planar (r.m.s. deviation for all non-H atoms = 0.0291 Å) and it forms dihedral angles of 5.63 (6)° with the C1-C6 and 10.20 (5)° with the C8-C13 rings respectively. Bond lengths and bond angles in the molecule are in a good agreement with those found in the related compounds (II) and (III). The conformation of the N—H group is *syn* to the —OH substituent in the benzoyl ring, which results in a short intramolecular N—H···O contact. In the crystal (Fig. 2), molecules are linked by strong O—H···O hydrogen bonds and weak C—H···O intermolecular contacts. Indeed, the O2-H20 at (x,y,z) acts as a hydrogen-bond donor to O1 atom of the carbonyl group at (x+1,+y,+z) and the C6-H6 acts as a hydrogen-bond donor to O2 atom of the hydroxyl group at (x-1,+y,+z). These interactions generate C(7) chains of molecules and $R_2^2(7)$ rings (See Fig. 2), running along [100]. Additionally, the molecules are linked by N—H···O interactions. N1-H1 acts as a hydrogen-bond donor to O2 atom of the hydroxyl group at (-x+1,-y,-z+1), forming inversion dimers (Fig. 3).

S2. Experimental

The title molecule was synthesized taking 0.100 g (0.812 mmol) of 2-amino-4-methylphenol dissolved in acetonitrile (10 mL), and then was added benzoyl chloride (0.100 mL, 0.860 mmol). The solution was placed under reflux and constant stirring for 3 hours at 150°C. The solid was filtered and recrystallized from methanol. The solvent was evaporated at room temperature and pink crystals were obtained (m.p. 448 (1)K).

S3. Refinement

All H-atoms were positioned in geometrically idealized positions, C—H = 0.95 Å, and were refined using a riding-model approximation with $U_{\text{iso}}(\text{H})$ constrained to 1.2 times U_{eq} of the respective parent atom. H1 atom was found from the Fourier maps and its coordinates were refined freely.

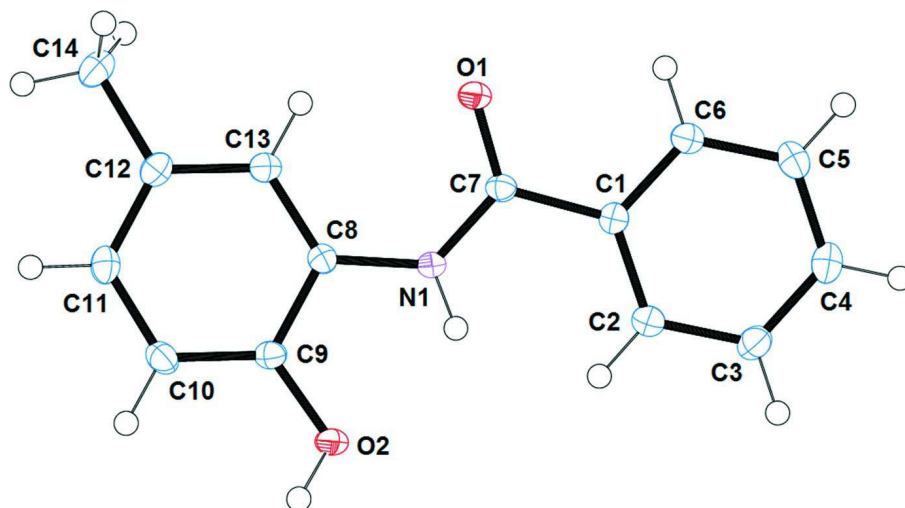


Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

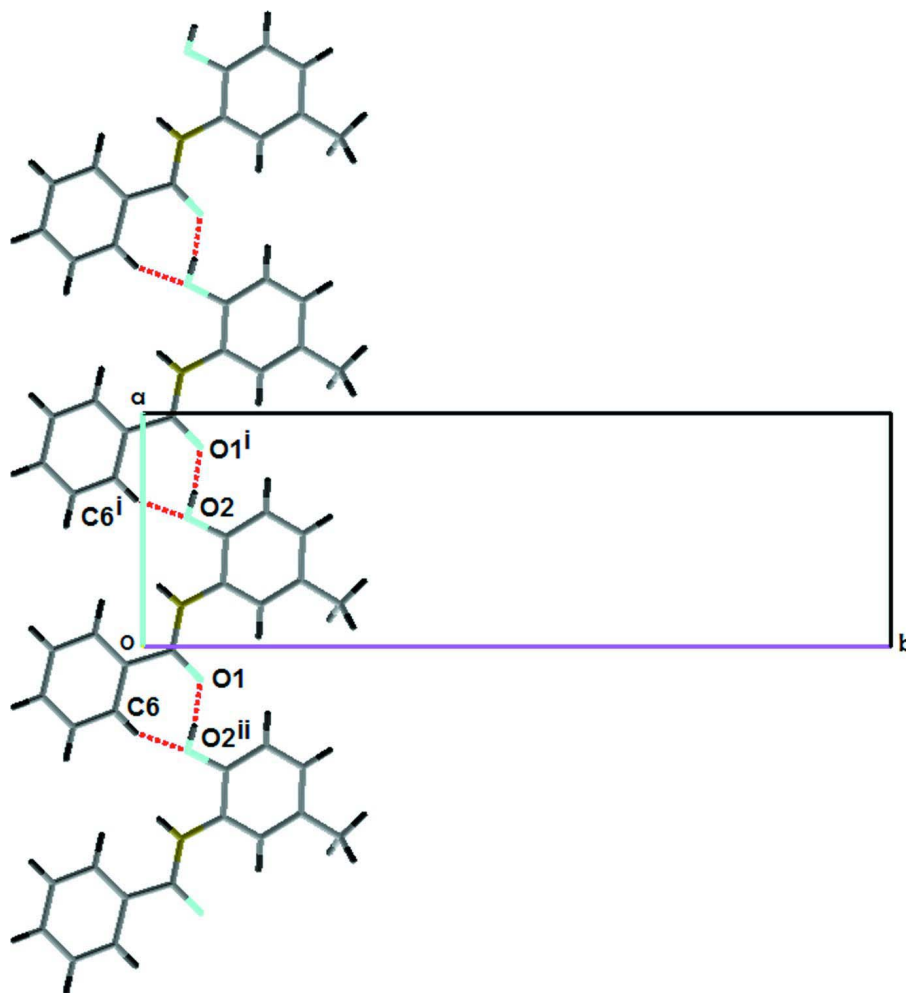


Figure 2

Part of the crystal structure of (I), showing the formation of C(7) chains of molecules along [100] [Symmetry codes: (i) $x + 1, +y, +z$; (ii) $x - 1, +y, +z$].

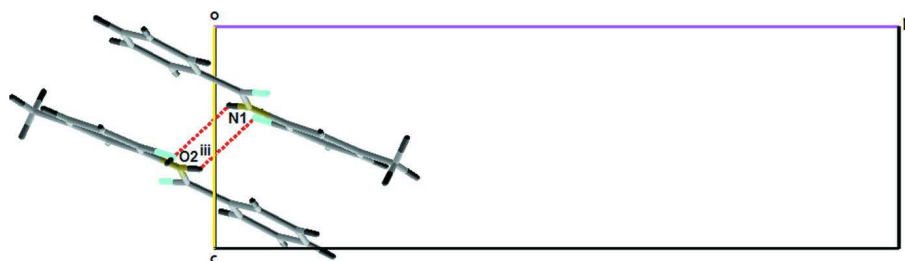


Figure 3

Part of the crystal structure of (I), showing the formation of dimers along [001]. [Symmetry codes: (iii) $-x + 1, -y, -z + 1$].

N-(2-Hydroxy-5-methylphenyl)benzamide

Crystal data

$C_{14}H_{13}NO_2$
 $M_r = 227.25$

Monoclinic, $P2_1/n$
 $a = 7.2263(3) \text{ \AA}$

$b = 21.7442$ (7) Å
 $c = 7.4747$ (3) Å
 $\beta = 110.280$ (5)°
 $V = 1101.69$ (8) Å³
 $Z = 4$
 $F(000) = 480$
 $D_x = 1.370$ Mg m⁻³
 Melting point: 448(1) K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 10254 reflections
 $\theta = 3.4$ – 29.4 °
 $\mu = 0.09$ mm⁻¹
 $T = 123$ K
 Block, pink
 $0.40 \times 0.35 \times 0.25$ mm

Data collection

Oxford Diffraction Gemini S CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 10254 measured reflections
 2795 independent reflections

2332 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 29.4$ °, $\theta_{\text{min}} = 3.4$ °
 $h = -9 \rightarrow 9$
 $k = -30 \rightarrow 30$
 $l = -9 \rightarrow 10$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.109$
 $S = 1.03$
 2795 reflections
 163 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: mixed
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.5141P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Special details

Experimental. The IR spectrum was recorded on a FT—IR SHIMADZU IR-Affinity-1 spectrophotometer. IR (KBr), cm⁻¹, 3395 (amide N-H); 3073 (Hydroxyl O-H), 1643 (amide, C=O); 1593 (C=C).

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	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.55117 (13)	0.05937 (4)	0.41236 (14)	0.0204 (2)
O1	-0.14306 (13)	0.07692 (4)	0.30232 (14)	0.0214 (2)
N1	0.17579 (15)	0.05132 (5)	0.36397 (15)	0.0166 (2)
C1	-0.07846 (17)	-0.02289 (5)	0.20522 (16)	0.0153 (2)
C2	0.05257 (19)	-0.06486 (6)	0.17363 (18)	0.0192 (3)
H2	0.1892	-0.0554	0.2136	0.023*
C3	-0.0159 (2)	-0.12053 (6)	0.08386 (19)	0.0222 (3)
H3	0.0743	-0.1490	0.0632	0.027*

C4	-0.2145 (2)	-0.13469 (6)	0.02438 (18)	0.0217 (3)
H4	-0.2607	-0.1728	-0.0371	0.026*
C5	-0.34605 (19)	-0.09320 (6)	0.05467 (19)	0.0224 (3)
H5	-0.4827	-0.1028	0.0136	0.027*
C6	-0.27845 (18)	-0.03785 (6)	0.14471 (18)	0.0193 (3)
H6	-0.3693	-0.0097	0.1656	0.023*
C7	-0.01844 (17)	0.03897 (5)	0.29485 (17)	0.0150 (2)
C8	0.26922 (17)	0.10733 (5)	0.43855 (17)	0.0149 (2)
C13	0.17867 (18)	0.15716 (6)	0.48997 (17)	0.0172 (3)
H13	0.0430	0.1547	0.4756	0.021*
C12	0.28408 (19)	0.21075 (6)	0.56243 (18)	0.0186 (3)
C11	0.48182 (19)	0.21370 (6)	0.58174 (18)	0.0202 (3)
H11	0.5548	0.2501	0.6301	0.024*
C10	0.57479 (18)	0.16395 (6)	0.53108 (18)	0.0195 (3)
H10	0.7104	0.1666	0.5452	0.023*
C9	0.46985 (17)	0.11075 (5)	0.46028 (17)	0.0157 (2)
C14	0.1817 (2)	0.26392 (6)	0.6182 (2)	0.0258 (3)
H141	0.2788	0.2956	0.6812	0.039*
H142	0.1168	0.2495	0.7059	0.039*
H143	0.0827	0.2813	0.5038	0.039*
H1	0.258 (2)	0.0217 (8)	0.357 (2)	0.031 (4)*
H20	0.658 (3)	0.0685 (9)	0.383 (3)	0.047 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0158 (4)	0.0173 (4)	0.0316 (5)	0.0009 (3)	0.0126 (4)	-0.0013 (4)
O1	0.0150 (4)	0.0177 (4)	0.0333 (5)	0.0005 (3)	0.0108 (4)	-0.0022 (4)
N1	0.0129 (5)	0.0147 (5)	0.0224 (5)	0.0010 (4)	0.0063 (4)	-0.0027 (4)
C1	0.0170 (6)	0.0148 (5)	0.0142 (5)	-0.0004 (4)	0.0056 (4)	0.0008 (4)
C2	0.0161 (6)	0.0200 (6)	0.0210 (6)	0.0009 (5)	0.0056 (5)	-0.0008 (5)
C3	0.0235 (7)	0.0188 (6)	0.0240 (6)	0.0027 (5)	0.0078 (5)	-0.0030 (5)
C4	0.0267 (7)	0.0172 (6)	0.0190 (6)	-0.0028 (5)	0.0051 (5)	-0.0012 (5)
C5	0.0186 (6)	0.0230 (6)	0.0238 (6)	-0.0047 (5)	0.0051 (5)	-0.0013 (5)
C6	0.0162 (6)	0.0197 (6)	0.0220 (6)	0.0008 (5)	0.0065 (5)	-0.0002 (5)
C7	0.0143 (5)	0.0156 (6)	0.0164 (5)	0.0010 (4)	0.0069 (4)	0.0014 (4)
C8	0.0145 (6)	0.0150 (5)	0.0146 (5)	-0.0003 (4)	0.0043 (4)	0.0009 (4)
C13	0.0161 (6)	0.0180 (6)	0.0180 (6)	0.0012 (4)	0.0065 (5)	-0.0001 (4)
C12	0.0227 (6)	0.0155 (6)	0.0178 (6)	0.0015 (5)	0.0073 (5)	0.0006 (4)
C11	0.0230 (7)	0.0149 (6)	0.0222 (6)	-0.0034 (5)	0.0071 (5)	0.0006 (5)
C10	0.0152 (6)	0.0195 (6)	0.0238 (6)	-0.0019 (5)	0.0069 (5)	0.0027 (5)
C9	0.0153 (6)	0.0156 (5)	0.0172 (6)	0.0022 (4)	0.0070 (5)	0.0022 (4)
C14	0.0297 (7)	0.0184 (6)	0.0308 (7)	0.0023 (5)	0.0125 (6)	-0.0044 (5)

Geometric parameters (Å, °)

O2—C9	1.3667 (14)	C5—H5	0.9500
O2—H20	0.89 (2)	C6—H6	0.9500

O1—C7	1.2369 (14)	C8—C13	1.3871 (16)
N1—C7	1.3440 (15)	C8—C9	1.4038 (16)
N1—C8	1.4103 (15)	C13—C12	1.3949 (17)
N1—H1	0.889 (18)	C13—H13	0.9500
C1—C2	1.3929 (17)	C12—C11	1.3870 (18)
C1—C6	1.3949 (17)	C12—C14	1.5074 (17)
C1—C7	1.4984 (16)	C11—C10	1.3933 (18)
C2—C3	1.3893 (17)	C11—H11	0.9500
C2—H2	0.9500	C10—C9	1.3838 (17)
C3—C4	1.3818 (19)	C10—H10	0.9500
C3—H3	0.9500	C14—H141	0.9800
C4—C5	1.3849 (19)	C14—H142	0.9800
C4—H4	0.9500	C14—H143	0.9800
C5—C6	1.3829 (18)		
C9—O2—H20	111.6 (12)	C13—C8—C9	119.61 (11)
C7—N1—C8	128.14 (10)	C13—C8—N1	125.23 (11)
C7—N1—H1	117.3 (11)	C9—C8—N1	115.16 (10)
C8—N1—H1	114.4 (11)	C8—C13—C12	120.90 (11)
C2—C1—C6	118.75 (11)	C8—C13—H13	119.5
C2—C1—C7	123.75 (11)	C12—C13—H13	119.5
C6—C1—C7	117.46 (10)	C11—C12—C13	118.90 (11)
C3—C2—C1	120.22 (12)	C11—C12—C14	121.51 (12)
C3—C2—H2	119.9	C13—C12—C14	119.59 (11)
C1—C2—H2	119.9	C12—C11—C10	120.82 (11)
C4—C3—C2	120.39 (12)	C12—C11—H11	119.6
C4—C3—H3	119.8	C10—C11—H11	119.6
C2—C3—H3	119.8	C9—C10—C11	120.07 (11)
C3—C4—C5	119.83 (12)	C9—C10—H10	120.0
C3—C4—H4	120.1	C11—C10—H10	120.0
C5—C4—H4	120.1	O2—C9—C10	123.73 (11)
C6—C5—C4	119.99 (12)	O2—C9—C8	116.55 (10)
C6—C5—H5	120.0	C10—C9—C8	119.71 (11)
C4—C5—H5	120.0	C12—C14—H141	109.5
C5—C6—C1	120.81 (12)	C12—C14—H142	109.5
C5—C6—H6	119.6	H141—C14—H142	109.5
C1—C6—H6	119.6	C12—C14—H143	109.5
O1—C7—N1	121.94 (11)	H141—C14—H143	109.5
O1—C7—C1	121.11 (11)	H142—C14—H143	109.5
N1—C7—C1	116.95 (10)		
C6—C1—C2—C3	-0.16 (18)	C7—N1—C8—C9	-166.27 (11)
C7—C1—C2—C3	-177.86 (11)	C9—C8—C13—C12	0.22 (18)
C1—C2—C3—C4	0.27 (19)	N1—C8—C13—C12	-179.91 (11)
C2—C3—C4—C5	-0.10 (19)	C8—C13—C12—C11	0.28 (18)
C3—C4—C5—C6	-0.2 (2)	C8—C13—C12—C14	-179.59 (12)
C4—C5—C6—C1	0.3 (2)	C13—C12—C11—C10	-0.44 (19)
C2—C1—C6—C5	-0.12 (18)	C14—C12—C11—C10	179.44 (12)

C7—C1—C6—C5	177.72 (11)	C12—C11—C10—C9	0.08 (19)
C8—N1—C7—O1	-5.48 (19)	C11—C10—C9—O2	-178.11 (11)
C8—N1—C7—C1	173.71 (10)	C11—C10—C9—C8	0.44 (18)
C2—C1—C7—O1	172.66 (12)	C13—C8—C9—O2	178.06 (11)
C6—C1—C7—O1	-5.06 (17)	N1—C8—C9—O2	-1.82 (15)
C2—C1—C7—N1	-6.54 (17)	C13—C8—C9—C10	-0.59 (17)
C6—C1—C7—N1	175.74 (11)	N1—C8—C9—C10	179.54 (11)
C7—N1—C8—C13	13.9 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O2	0.889 (18)	2.173 (16)	2.6153 (15)	110.0 (13)
N1—H1 \cdots O2 ⁱ	0.889 (18)	2.518 (17)	3.1928 (14)	133.1 (14)
O2—H20 \cdots O1 ⁱⁱ	0.89 (2)	1.75 (2)	2.6390 (12)	171.3 (19)
C6—H6 \cdots O2 ⁱⁱⁱ	0.95	2.59	3.4197 (15)	146

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x+1, y, z$; (iii) $x-1, y, z$.