Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Hexyloxybenzamide

Huey Chong Kwong, Mohamad Zaki Ab. Rahman, Mohamed Ibrahim Mohamed Tahir and Sidik Silong*

Department of Chemistry, Faculty of Science, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
Correspondence e-mail: sidik@science.upm.edu.my

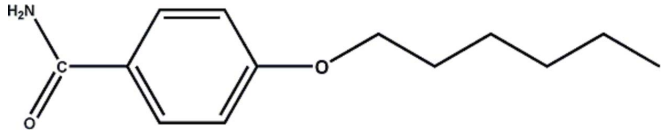
Received 29 December 2010; accepted 24 January 2011

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.095; data-to-parameter ratio = 14.5.

In the title compound, $C_{13}H_{19}NO_2$, the dihedral angle between the benzene ring and the plane throught the non-H atoms of the amide group is 29.3 (1)°. The benzene ring and the alkane carbon skeleton plane are twisted slightly with respect to each other [5.40 (5)°]. In the crystal, molecules are oriented with the amide groups head-to-head, forming N—H···O hydrogen-bonded dimers. The dimers are connected by further N—H···O hydrogen bonds into a ladder-like motif along the b axis.

Related literature

For standard bond lengths, see Allen *et al.* (1987). For related structures, see: Merz (2002); Jones *et al.* (2002); Pagola & Stephens (2009); Boese *et al.* (1999). For related experiments on the hydrolysis of nitrites, see: Gallardo & Begnini (1995); Pala Wilgus *et al.* (1995).



Experimental

Crystal data

$C_{13}H_{19}NO_2$
M_r = 221.30
Monoclinic, $P2_1/n$
a = 12.5507 (2) Å
b = 5.16441 (9) Å
c = 18.9322 (3) Å
β = 91.4702 (16)°
V = 1226.72 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.64 mm^{-1}
T = 150 K
0.31 × 0.08 × 0.05 mm

Data collection

Oxford Diffraction Gemini E diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*: Oxford Diffraction, 2006)
T_{min} = 0.894, T_{max} = 1.000
13258 measured reflections
2355 independent reflections
2165 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.01
2101 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N10—H101···O9^{i}	0.90	2.14	3.0153 (18)	164
N10—H102···O9^{ii}	0.90	2.04	2.9401 (18)	174

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *CRYSTALS*.

The author would like to acknowledge the Ministry of Science, Technology and Innovation (MOSTI) Malaysia for funding (Research Grant No. 04–01–04-SF0144 and 05–02-10–0934RU).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2300).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Prpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Boese, R., Weiss, H.-C. & Blaeser, D. (1999). *Angew. Chem. Int. Ed.* **38**, 988–992.
Gallardo, H. & Begnini, I. M. (1995). *Mol. Cryst. Liq. Cryst. Sci. Technol.* **258**, 85–94.
Jones, P. G., Thönnessen, H., Schmutzler, R. & Fischer, A. K. (2002). *Acta Cryst.* E**58**, o1436–o1438.
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.
Merz, K. (2002). *Acta Cryst.* E**58**, o450–o451.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Pagola, S. & Stephens, P. W. (2009). *Acta Cryst.* C**65**, o583–o586.
Pala Wilgus, C., Downing, S., Molitor, E., Bains, S., Pagni, R. M. & Kabalka, G. W. (1995). *Tetrahedron Lett.* **36**, 3469–3472

supporting information

Acta Cryst. (2011). E**67**, o612 [doi:10.1107/S1600536811003096]

4-Hexyloxybenzamide

Huey Chong Kwong, Mohamad Zaki Ab. Rahman, Mohamed Ibrahim Mohamed Tahir and Sidik Silong

S1. Comment

Bond distance and angles in the titled compound (I), 4-(hexyloxy)benzamide (Fig.1) are in normal range (Allen *et al.* 1987) and are comparable with those in closely related structure which was determined by single-crystal X-ray diffraction method (Merz, 2002; Jones *et al.*, 2002). On the other hand, bond distances at ether and amide group differ from those in the structure of 2-ethoxybenzamide (Pagola & Stephens, 2009). The benzene ring with O1 and C8 is nearly planar (r.m.s deviation of 0.0004 Å), whereas C7 is 0.019Å out of this plane. The dihedral angle between the benzene ring and the amide group plane is 29.3° [20.1° in *p*-nitrobenzamide (Jones *et al.*, 2002)].In the molecular structure of (I) alkane carbon skeleton is not coplanar with the aromatic ring; the dihedral angle between the benzene ring and the alkane carbon skeleton is 5.40 (1)°. The torsion angles along the alkane carbon skeleton increase (torsion angle of C11—C14=174.1°, C12—C15=175.9°, and C13—C16=178.2°). The arrangement observed here is slightly different from n-alkanes which contain a planar zigzag carbon skeleton. However, the mean C(H3)—C(H2) and C(H2)—C(H2) distance, and C(H3)—C(H2)—C and C(H2)—C(H2)—C angles, are in agreement with those determined for n-alkanes [1.521 (1)Å and 112.8 (1)° and 113.5 (1)°, respectively; Boese *et al.*, 1999]. In the crystal structure, the amide groups are oriented head-to-head forming N10—H102···O9 hydrogen bond at (-*x*, -*y* + 1, -*z* + 1) [the N···O distance is 1.941 Å] to generate a hydrogen bond dimer. These dimers are further linked together by N10—H101···O9 hydrogen bonding at (*x*,*y* + 1, *z*) [N···O distance is 3.106 Å] generating a ladder-like motif along the *b* axis (Table 1, Figs. 2 and 3) (Pagola & Stephens, 2009).

S2. Experimental

Attempts to crystallize 1,2-bis(4-heptylbenzylidene)hydrazine by liquid diffusion method from n-buthanol and water led to crystals of the title compound, presumably due to slow hydrolysis by supervenient of water (Gallardo & Begnini, 1995; Pala Wilgus *et al.*, 1995).

S3. Refinement

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, N—H in the range 0.86–0.90 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

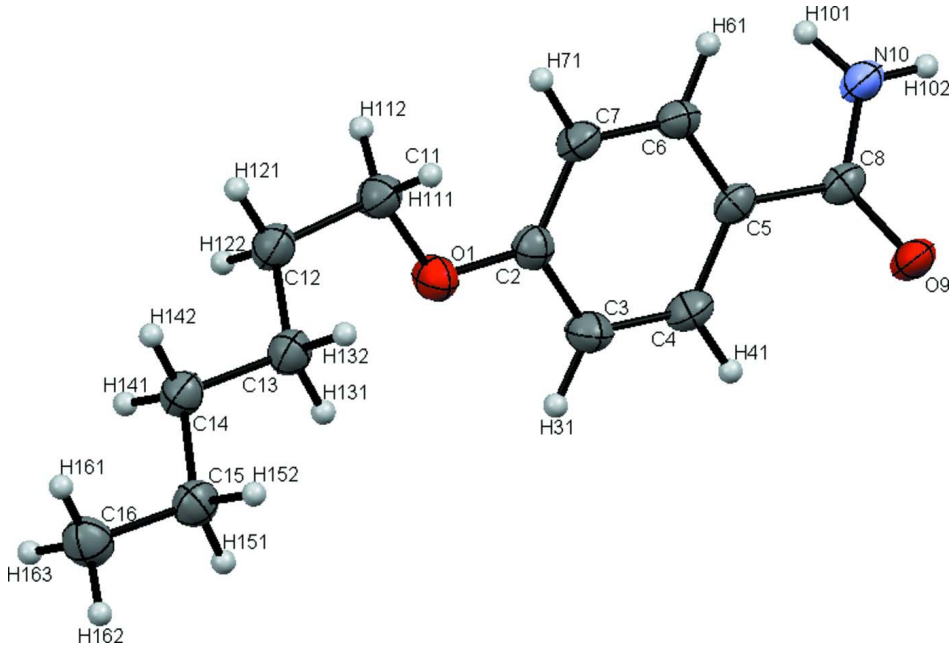


Figure 1

Molecular structure of (I) with arom numbering and displacement ellipsoids at the 50% probablity level.

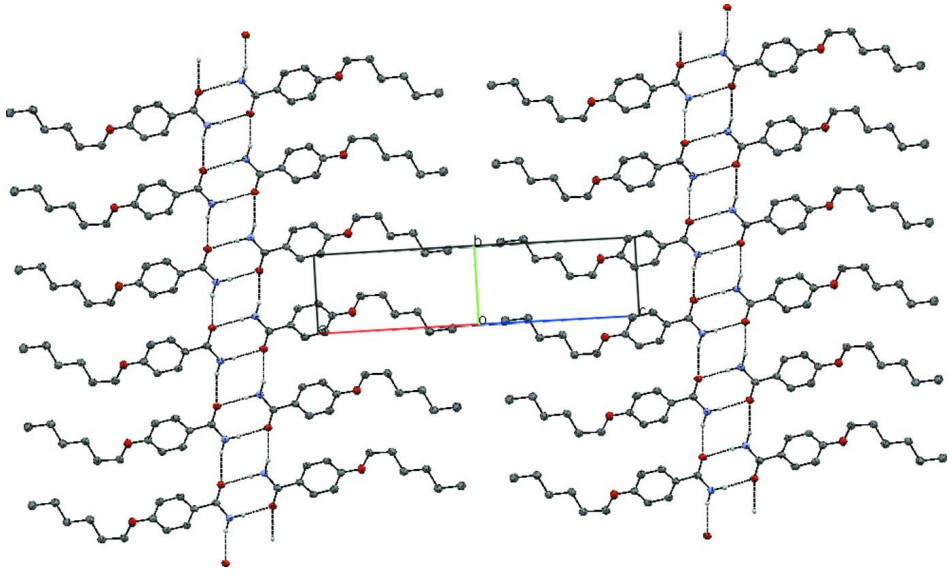


Figure 2

The hydrogen-bonded ladder-like motif (dashed lines) extends along *b*-axis.

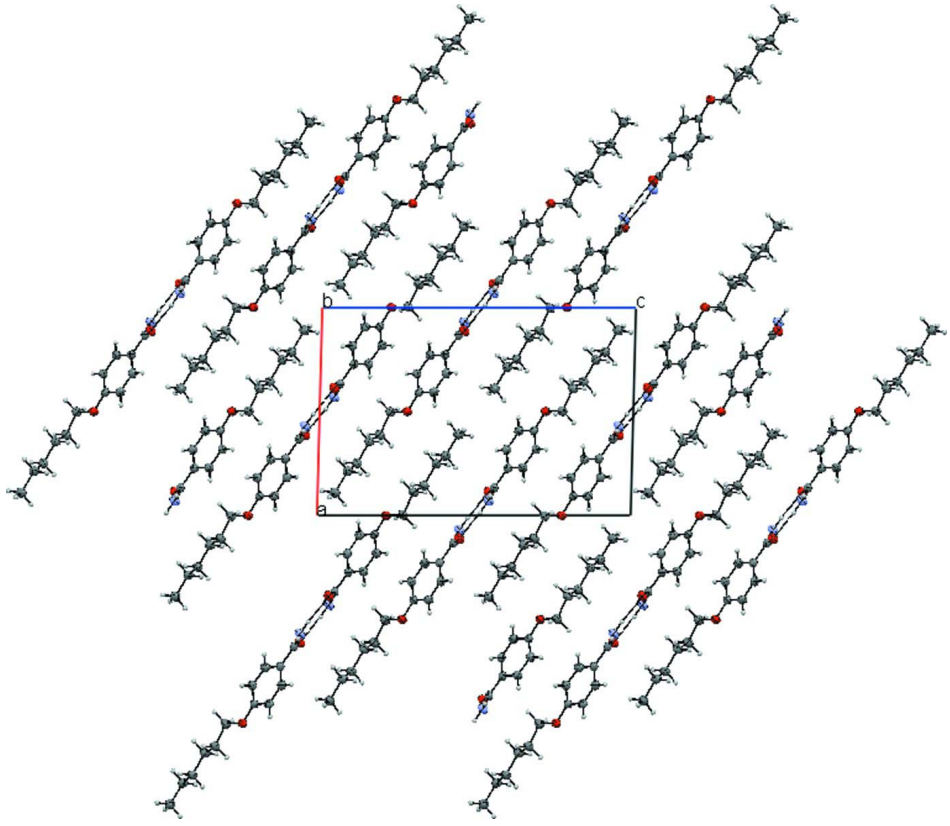


Figure 3

Packing diagram of the title compound viewed along the *a* axis; hydrogen bond are shown as dashed lines.

4-Hexyloxybenzamide

Crystal data

$C_{13}H_{19}NO_2$
$M_r = 221.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.5507\ (2)$ Å
$b = 5.16441\ (9)$ Å
$c = 18.9322\ (3)$ Å
$\beta = 91.4702\ (16)°$
$V = 1226.72\ (4)$ Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.198$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 8720 reflections
$\theta = 3.5–70.8°$
$\mu = 0.64$ mm^{-1}
$T = 150$ K
Needle-like, colourless
0.31 × 0.08 × 0.05 mm

Data collection

Oxford Diffraction Gemini E diffractometer
Radiation source: sealed x-ray tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: multi-scan (*CrysAlis PRO*: Oxford Diffraction, 2006)
$T_{min} = 0.894$, $T_{max} = 1.000$
13258 measured reflections
2355 independent reflections
2165 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 71.0°$, $\theta_{min} = 4.2°$
$h = -15\rightarrow13$
$k = -6\rightarrow6$
$l = -21\rightarrow23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.095$
$S = 1.01$
2101 reflections
145 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2 + 0.41P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.0002608$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Refinement. For this compound, 13258 reflections were measured and collected during the refinement. However after merging the symmetry equivalent reflections, there were only 2355 independent reflections. Further 254 more reflections were filtered, as sigma cutoff was set at 3.0 and (sin theta x 2)set to >0.01 (to eliminate reflection measured near the vicinity of beam stop) therefore reduced the number of reflection to 2101 which were used in the Refinement.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.50007 (7)	0.75339 (18)	0.27977 (5)	0.0339
C2	0.40797 (9)	0.7236 (2)	0.31525 (6)	0.0276
C3	0.40594 (10)	0.5166 (2)	0.36254 (7)	0.0327
C4	0.31610 (10)	0.4686 (2)	0.40089 (6)	0.0302
C5	0.22742 (9)	0.6304 (2)	0.39492 (6)	0.0258
C6	0.23026 (10)	0.8374 (2)	0.34797 (6)	0.0283
C7	0.31893 (10)	0.8824 (2)	0.30718 (6)	0.0292
C8	0.13215 (9)	0.5718 (2)	0.43773 (6)	0.0265
O9	0.11152 (7)	0.34604 (16)	0.45547 (5)	0.0322
N10	0.07033 (9)	0.7708 (2)	0.45501 (6)	0.0326
C11	0.50247 (10)	0.9496 (3)	0.22591 (7)	0.0334
C12	0.61157 (10)	0.9498 (2)	0.19414 (7)	0.0337
C13	0.64375 (10)	0.6928 (2)	0.16146 (7)	0.0321
C14	0.74928 (10)	0.7091 (2)	0.12345 (7)	0.0314
C15	0.78664 (10)	0.4500 (3)	0.09497 (7)	0.0339
C16	0.89010 (11)	0.4699 (3)	0.05528 (7)	0.0392
H31	0.4688	0.4087	0.3672	0.0424*
H41	0.3139	0.3171	0.4318	0.0397*
H61	0.1691	0.9533	0.3429	0.0368*
H71	0.3177	1.0309	0.2744	0.0385*
H112	0.4882	1.1252	0.2475	0.0433*
H111	0.4461	0.9082	0.1885	0.0424*
H122	0.6652	1.0029	0.2319	0.0438*
H121	0.6111	1.0874	0.1576	0.0426*
H131	0.6479	0.5555	0.1986	0.0402*
H132	0.5859	0.6413	0.1263	0.0417*
H141	0.8049	0.7782	0.1575	0.0417*
H142	0.7433	0.8391	0.0847	0.0407*
H151	0.7949	0.3232	0.1343	0.0435*

H152	0.7299	0.3781	0.0628	0.0445*
H162	0.9124	0.2983	0.0389	0.0627*
H163	0.9472	0.5385	0.0870	0.0619*
H161	0.8823	0.5875	0.0142	0.0630*
H101	0.0938	0.9339	0.4498	0.0433*
H102	0.0124	0.7437	0.4808	0.0432*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0292 (4)	0.0377 (5)	0.0350 (5)	0.0032 (4)	0.0051 (4)	0.0078 (4)
C2	0.0281 (6)	0.0268 (6)	0.0279 (6)	−0.0015 (5)	0.0011 (4)	−0.0021 (5)
C3	0.0319 (6)	0.0296 (6)	0.0365 (7)	0.0059 (5)	0.0000 (5)	0.0042 (5)
C4	0.0367 (7)	0.0238 (6)	0.0300 (6)	0.0010 (5)	0.0007 (5)	0.0046 (5)
C5	0.0312 (6)	0.0199 (5)	0.0264 (5)	−0.0024 (4)	0.0004 (4)	−0.0026 (4)
C6	0.0317 (6)	0.0209 (6)	0.0325 (6)	0.0024 (5)	0.0019 (5)	0.0006 (5)
C7	0.0346 (6)	0.0220 (6)	0.0311 (6)	−0.0002 (5)	0.0029 (5)	0.0039 (5)
C8	0.0331 (6)	0.0216 (6)	0.0248 (5)	−0.0019 (5)	0.0001 (5)	−0.0006 (4)
O9	0.0390 (5)	0.0201 (4)	0.0378 (5)	−0.0009 (3)	0.0095 (4)	0.0028 (3)
N10	0.0382 (6)	0.0204 (5)	0.0399 (6)	−0.0006 (4)	0.0136 (5)	0.0011 (4)
C11	0.0356 (7)	0.0278 (6)	0.0373 (7)	0.0011 (5)	0.0070 (5)	0.0042 (5)
C12	0.0353 (7)	0.0279 (6)	0.0382 (7)	−0.0037 (5)	0.0071 (5)	0.0001 (5)
C13	0.0316 (6)	0.0277 (6)	0.0370 (7)	−0.0031 (5)	0.0030 (5)	−0.0013 (5)
C14	0.0335 (6)	0.0271 (6)	0.0335 (6)	−0.0027 (5)	0.0024 (5)	−0.0002 (5)
C15	0.0335 (7)	0.0292 (7)	0.0391 (7)	−0.0018 (5)	0.0013 (5)	−0.0039 (5)
C16	0.0368 (7)	0.0393 (8)	0.0415 (7)	0.0014 (6)	0.0031 (6)	−0.0071 (6)

Geometric parameters (Å, º)

O1—C2	1.3605 (14)	C11—H112	1.013
O1—C11	1.4384 (15)	C11—H111	1.011
C2—C3	1.3954 (17)	C12—C13	1.5233 (17)
C2—C7	1.3915 (17)	C12—H122	1.007
C3—C4	1.3792 (17)	C12—H121	0.992
C3—H31	0.968	C13—C14	1.5258 (17)
C4—C5	1.3943 (17)	C13—H131	0.998
C4—H41	0.978	C13—H132	1.007
C5—C6	1.3912 (16)	C14—C15	1.5216 (17)
C5—C8	1.4926 (16)	C14—H141	1.003
C6—C7	1.3904 (17)	C14—H142	0.996
C6—H61	0.976	C15—C16	1.5203 (18)
C7—H71	0.986	C15—H151	0.995
C8—O9	1.2423 (14)	C15—H152	0.997
C8—N10	1.3338 (15)	C16—H162	0.982
N10—H101	0.898	C16—H163	0.989
N10—H102	0.898	C16—H161	0.990
C11—C12	1.5096 (17)		

C2—O1—C11	117.58 (9)	C11—C12—C13	114.45 (10)
O1—C2—C3	115.66 (11)	C11—C12—H122	108.3
O1—C2—C7	124.71 (11)	C13—C12—H122	110.2
C3—C2—C7	119.63 (11)	C11—C12—H121	106.8
C2—C3—C4	120.28 (11)	C13—C12—H121	109.7
C2—C3—H31	118.1	H122—C12—H121	106.9
C4—C3—H31	121.6	C12—C13—C14	112.69 (10)
C3—C4—C5	120.75 (11)	C12—C13—H131	110.0
C3—C4—H41	119.7	C14—C13—H131	110.0
C5—C4—H41	119.5	C12—C13—H132	107.7
C4—C5—C6	118.62 (11)	C14—C13—H132	108.8
C4—C5—C8	118.89 (10)	H131—C13—H132	107.5
C6—C5—C8	122.47 (11)	C13—C14—C15	113.43 (10)
C5—C6—C7	121.16 (11)	C13—C14—H141	108.4
C5—C6—H61	120.1	C15—C14—H141	109.0
C7—C6—H61	118.8	C13—C14—H142	109.6
C2—C7—C6	119.50 (11)	C15—C14—H142	110.5
C2—C7—H71	121.8	H141—C14—H142	105.7
C6—C7—H71	118.6	C14—C15—C16	112.97 (11)
C5—C8—O9	120.85 (10)	C14—C15—H151	109.9
C5—C8—N10	117.12 (10)	C16—C15—H151	109.9
O9—C8—N10	122.03 (11)	C14—C15—H152	108.8
C8—N10—H101	120.1	C16—C15—H152	109.2
C8—N10—H102	119.9	H151—C15—H152	105.8
H101—N10—H102	118.5	C15—C16—H162	110.4
O1—C11—C12	108.58 (10)	C15—C16—H163	109.8
O1—C11—H112	109.7	H162—C16—H163	107.9
C12—C11—H112	109.4	C15—C16—H161	111.2
O1—C11—H111	108.7	H162—C16—H161	109.2
C12—C11—H111	110.3	H163—C16—H161	108.3
H112—C11—H111	110.1		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H101···O9^{i}	0.90	2.14	3.0153 (18)	164
N10—H102···O9^{ii}	0.90	2.04	2.9401 (18)	174

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