Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of (*E*)-furan-2-carbaldehyde *O*-benzoyloxime

Yousef M. Hijji,[a]* Rajeesha Rajan,[a] Said Mansour[b] and Hamdi Ben Yahia[b]*

[a]Department of Chemistry and Earth Sciences, Qatar University, PO Box 2713, Doha, Qatar, and [b]Qatar Environment and Energy Research Institute, Hamad Bin Khalifa University, Qatar Foundation, PO Box 34110, Doha, Qatar. *Correspondence e-mail: yousef.hijji@qu.edu.qa, hyahia@qf.org.qa

Received 14 July 2017
Accepted 4 August 2017

Edited by T. N. Guru Row, Indian Institute of Science, India

Keywords: crystal structure; oxime; 2-furan-aldoxime; benzoyloxime ester; hydrogen bonding.

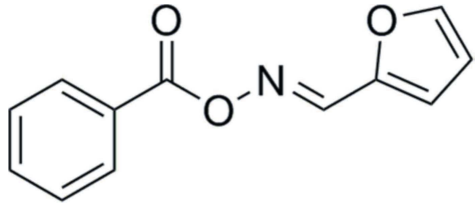
CCDC reference: 1549733

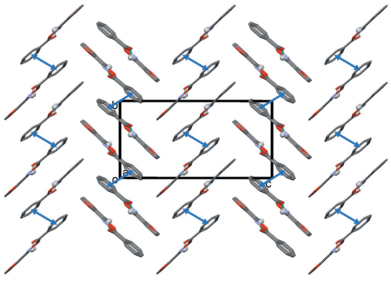
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $C_{12}H_9NO_3$, the benzoate and furan rings are almost coplanar, making a dihedral angle of 11.68 (9)°. The twist angle between the –COO group and the benzene ring is only 2.79 (16)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming chains along [100]. The molecules stack in a herringbone fashion and inversion-related chains are linked by offset π–π interactions [intercentroid distance = 3.931 (1) Å], forming ribbons propagating along the *a*-axis direction.

1. Chemical context

Oxime esters have shown potencies for inhibiting lipoprotein-associated phospholipase A2 (Lp-PLA2) activity. Their derivatives are used for the prevention and treatment of cardiovascular disease (Jeong *et al.*, 2013, 2006). These compounds are good antioxidants and are used in pharmaceutical compositions for their anti-microbial activity (Liu *et al.*, 2008; Harini *et al.*, 2012; Ahluwalia *et al.*, 2017). In view of this interest, we have synthesized the title oxime ester derivative and report herein on its crystal structure.





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. An intramolecular short contact (C8—H8···O2) is present (Table 1), which may prevent the –COO group from tilting, since the twist angle between the –C6/O2/O3 unit and the benzene ring (C7–C12) is only 2.79 (16)°. This also might be the reason why the molecule is almost planar. The dihedral angle between the furan (O1/C1–C4) ring and the benzene ring is 11.68 (9)°. The C6—O2 and C6═O3 distances of 1.352 (2) and 1.195 (2) Å, respectively, are typical values for single and double C-O bonds. This overall geometry is very similar to that observed for *E*-benzaldehyde *O*-benzoyloxime (Altinbas *et al.*, 2004). Within the five-membered furan ring, the interatomic O1—C1 and O1—C4 distances of 1.369 (2)

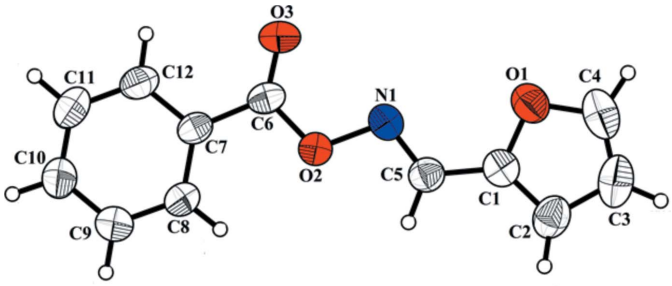


Figure 1
View of the molecular structure of the title compound, with the atom labelling and 50% probability displacement ellipsoids.

and 1.367 (2) Å, respectively, are typical values for O—Csp^2 bonds. The short C4—C3 and C1—C2 bond lengths of 1.324 (4) and 1.347 (3) Å, respectively, and the stretched C2—C3 bond distance of 1.408 (2) Å are typical values observed for double C═C and single C—C bonds, respectively. The –C5/N1/O2 group is twisted by 4.40 (13) ° with respect to the furan ring. The N1—O2 distance of 1.444 (1) Å is only slightly longer than reported in other oxime compounds (Wetherington & Moncrief, 1973), whereas the C═N—O angle of 106.73 (11)° is slightly smaller.

3. Supramolecular features

In the crystal, molecules are linked by C—H···O hydrogen bonds, forming chains along the *a*-axis direction (Table 1 and Fig. 2). The molecules stack in a herringbone fashion and inversion-related chains are linked by offset π–π interactions [$Cg1\cdots Cg1^{i}$ = 3.931 (1) Å, interplanar distance = 3.574 (1) Å, slippage = 1.64 Å, α = 0.03 (7)°, *Cg*1 is the centroid of the benzene ring (C7–C12); symmetry code: (i) $-x+1, -y+2, -z$], forming ribbons propagating along the *a*-axis direction (Fig. 3).

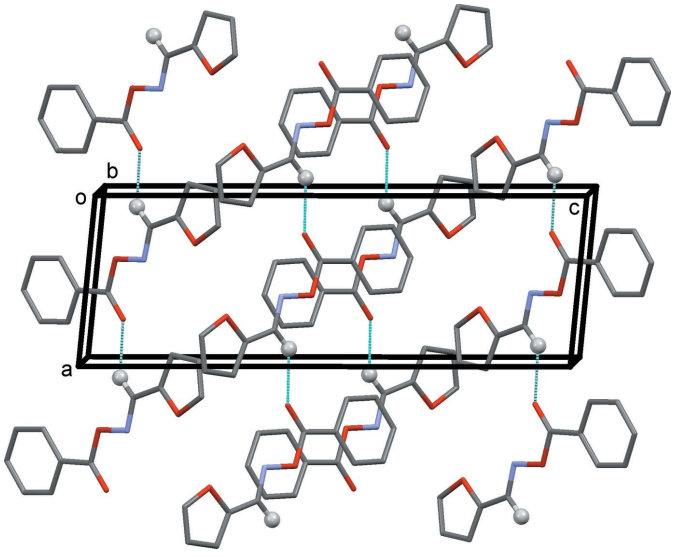


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The C—H···O hydrogen bonds, linking molecules to form chains along [100], are shown as dashed lines [see Table 1; only H atom H5 (grey ball) has been included].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2	0.93 (2)	2.384 (13)	2.724 (2)	102 (1)
C5—H5···O3^{i}	0.97 (2)	2.312 (16)	3.159 (2)	145 (1)

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.38, update May 2017; Groom *et al.*, 2016) for the substructure furan-2-carbaldehyde oxime gave 20 hits, while for substructure formaldehyde *O*-benzoyloxime there were 24 hits. The O—N distances vary from *ca* 1.38 to 1.45 Å, while the N═C distances vary from *ca* 1.25 to 1.32 Å. In the title compound, these distances are N1—O2 = 1.444 (1) Å and N1═C5 is 1.270 (2) Å, within the limits observed. In the majority of the formaldehyde *O*-benzoyloxime structures, the dihedral angle between the plane of the –COO group and the benzene ring is <10 °. In the title compound, this dihedral angle is 2.79 (16)°.

5. Synthesis and crystallization

Synthesis of 2-furanaldoxime: A mixture of 5.0 g of furfuraldehyde (without further purification), 1.5 equiv. of $NH_2OH{\cdot}HCl$ and 1 mmol of pyridine was stirred for 3 h at rt until the $NH_2OH{\cdot}HCl$ was completely solubilized. The reaction mixture was then quenched in water and the furanaldoxime precipitated out. This solid was filtered and recrystallized from diethyl ether to give colourless needle-like crystals (yield 4.268 g, 74%; m.p. 349–351 K). FT–IR spectrum showed two peaks at 3166 and 1634 cm^{-1}. Elemental analysis:

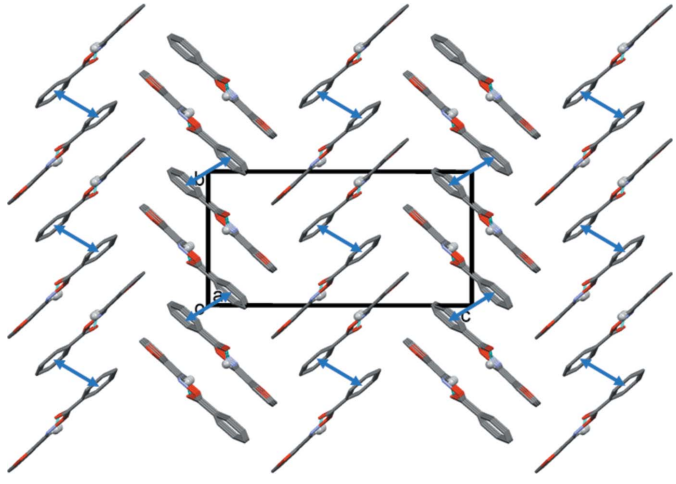


Figure 3
A view along the *a* axis of the crystal packing of the title compound. The offset π–π interactions are shown as blue double arrows, and only H atom H5 (grey ball) has been included.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_9NO_3$
M_r	215.2
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	6.3414 (3), 9.1268 (5), 18.1423 (9)
β (°)	95.634 (2)
V (Å^3)	1044.94 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.1
Crystal size (mm)	0.19 × 0.06 × 0.04
Data collection	
Diffractometer	D8 venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.87, 0.89
No. of measured, independent and observed [$I > 3\sigma(I)$] reflections	19019, 2480, 1245
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F > 3\sigma(F)]$, $wR(F)$, S	0.037, 0.101, 1.05
No. of reflections	2480
No. of parameters	182
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.19

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SIR2002* (Burla *et al.* 2003), *JANA2006* (Petricek *et al.*, 2014), *DIAMOND* (Brandenburg & Berndt, 1999) and *Mercury* (Macrae *et al.*, 2008).

analysis calculated for $C_5H_5NO_2$ (111.10 g mol^{-1}): C, 54.05; H, 4.54; N, 12.61; O, 28.80%. Found: C, 53.13; H, 4.45; N, 12.99; O, 29.43%. ^{1}H NMR (DMSO-d_6): δ (ppm): 6.64 (*dd*, J = 3.42Hz, 0.49 Hz, 1H), 7.20 (*d*, J = 3.42Hz, 1H), 7.52 (*s*, 1H), 7.76 (*s*, 1H), 11.80 (*s*, 1H). ^{13}C NMR (DMSO-d_6): δ (ppm) = 145.85, 143.80, 135.92, 116.89, 112.67.

Preparation of the *O*-benzoyl ester of furanaldoxime: Benzoyl chloride (5.01 mmol) was added dropwise under stirring to 4.55 mmol of furanaldoxime. Since the reaction was vigorous and exothermic the mixture was placed in an ice bath for 30 min. The reaction mixture was then quenched in ice–water, and then extracted with EtOAc. The organic layer was separated and washed with 1*M* NaOH solution to remove the benzoic acid and HCl that had formed as by products. The EtOAc layer was passed through anhydrous Na_2SO_4 and dried *in vacuo* to give the title compound as a light-brown solid (0.9806 g). Recrystallization of the title compound from ethanol–EtOAc gave colourless needle-like crystals (yield 50%, m.p. 410–412 K). Elemental analysis: analysis calculated for $C_{12}H_9NO_3$ (215.20 g mol^{-1}): C, 66.97; H, 4.22; N, 6.51; O, 22.30%. Found: C, 67.00; H, 4.19; N, 6.40; O, 22.41%. ^{1}H NMR (DMSO-d_6): δ (ppm): 6.74–6.75 (*dd*, J = 3.67Hz,1.96Hz, 1H), 7.18 (*d*, J = 3.42Hz, 1H), 7.60 (*t*, J = 8.04 Hz, 2H), 7.73 (*t*, J = 7.58Hz, 1H), 8.01 (*s*, 1H), 8.07 (*dd*, J = 8.56 Hz,1.22 Hz 2H), 8.82 (*s*, 1H). ^{13}C NMR (DMSO-d_6): δ ppm: 163.55, 148.05, 147.65, 145.28, 134.35, 129.77, 129.48, 128.52, 119.14, 113.07.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were located from difference-Fourier maps and freely refined.

Acknowledgements

We are grateful to Mr Ahmed Abdelsalam Ali Easa of the Central Laboratory Unit, Qatar University, for the elemental analysis, and to Mr Ziad Sarah from the American University of Sharjah for measuring the NMR data.

Funding information

Funding for this research was provided by: Qatar National Research Fund (award No. NPRP-7-495-1-094).

References

Ahluwalia, V., Kumar, J., Rana, V. S., Singh, R., Sati, O. P., Walia, S. & Garg, N. (2017). *Toxicol. Environ. Chem.* **99**, 1–9.
Altinbas, O., Dondas, H. A., Arslan, H., Kulcu, N. & Killner, C. (2004). *Z. Kristallogr. New Cryst. Struct.* **219**, 379.
Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2015). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Harini, S. T., Kumar, H. V., Rangaswamy, J. & Naik, N. (2012). *Bioorg. Med. Chem. Lett.* **22**, 7588–7592.
Jeong, T. S., Lee, W. S., Jeong, H. J., Park, Y. D., Han, J. M., Kim, H. C., Moon, O. S. & Won, Y. S. (2013). Google patent.
Jeong, H. J., Park, Y.-D., Park, H.-Y., Jeong, I. Y., Jeong, T.-S. & Lee, W. S. (2006). *Bioorg. Med. Chem. Lett.* **16**, 5576–5579.
Liu, X. H., Zhi, L. P., Song, B. A. & Xu, H. L. (2008). *Chem. Res. Chin. Univ.* **24**, 454–458.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Petricek, V., Dusek, M. & Palatinus, L. (2014). *Z. Kristallogr.* **229**, 345–352.
Wetherington, J. B. & Moncrief, J. W. (1973). *Acta Cryst.* B**29**, 1520–1525.

supporting information

Acta Cryst. (2017). E**73**, 1326-1328 [https://doi.org/10.1107/S2056989017011562]

Crystal structure of (*E*)-furan-2-carbaldehyde *O*-benzoyloxime

Yousef M. Hijji, Rajeesha Rajan, Said Mansour and Hamdi Ben Yahia

Computing details

Data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT* (Bruker, 2015); program(s) used to solve structure: *SIR2002* (Burla *et al.* 2003); program(s) used to refine structure: JANA2006 (Petricek *et al.*, 2014); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: JANA2006 (Petricek *et al.*, 2014).

(*E*)-(Furan-2-ylmethylidene)amino benzoate

Crystal data

$C_{12}H_9NO_3$
$M_r = 215.2$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.3414$ (3) Å
$b = 9.1268$ (5) Å
$c = 18.1423$ (9) Å
$\beta = 95.634$ (2)°
$V = 1044.94$ (9) Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.368$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 19019 reflections
$\theta = 2.3$–27.9°
$\mu = 0.1$ mm^{-1}
$T = 293$ K
Needle, colourless
0.19 × 0.06 × 0.04 mm

Data collection

D8 venture diffractometer
Radiation source: X-ray tube
ω and π scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
$T_{min} = 0.87$, $T_{max} = 0.89$
19019 measured reflections
2480 independent reflections
1245 reflections with $I > 3\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.3°$
$h = -7 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F > 3\sigma(F)] = 0.037$
$wR(F) = 0.101$
$S = 1.05$
2480 reflections
182 parameters
0 restraints
0 constraints
All H-atom parameters refined
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.001936I^2)$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: B–C type 1 Gaussian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 8600 (1100)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.30081 (17)	0.35070 (10)	0.20660 (6)	0.0583 (4)
O2	0.40886 (16)	0.65844 (10)	0.04205 (5)	0.0509 (4)
O3	0.76174 (18)	0.66965 (13)	0.06946 (6)	0.0708 (5)
N1	0.4062 (2)	0.55510 (12)	0.10223 (6)	0.0514 (5)
C1	0.1492 (3)	0.42685 (14)	0.16340 (8)	0.0476 (5)
C2	−0.0453 (3)	0.39050 (16)	0.18114 (10)	0.0599 (7)
H2	−0.177 (3)	0.4294 (17)	0.1589 (9)	0.071 (5)*
C3	−0.0152 (4)	0.28632 (18)	0.23844 (10)	0.0695 (8)
H3	−0.120 (3)	0.2372 (18)	0.2617 (9)	0.076 (5)*
C4	0.1916 (4)	0.26537 (18)	0.25172 (10)	0.0682 (8)
H4	0.279 (3)	0.2027 (17)	0.2837 (9)	0.074 (5)*
C5	0.2128 (3)	0.52772 (15)	0.10928 (8)	0.0468 (5)
H5	0.099 (3)	0.5716 (15)	0.0768 (8)	0.060 (4)*
C6	0.6056 (2)	0.70645 (15)	0.03182 (8)	0.0448 (5)
C7	0.6002 (2)	0.81086 (13)	−0.03072 (7)	0.0402 (5)
C8	0.4156 (3)	0.84781 (16)	−0.07375 (8)	0.0505 (6)
H8	0.290 (2)	0.8040 (15)	−0.0630 (7)	0.058 (4)*
C9	0.4214 (3)	0.94724 (17)	−0.13070 (10)	0.0600 (7)
H9	0.295 (3)	0.9700 (17)	−0.1605 (9)	0.073 (5)*
C10	0.6096 (3)	1.01023 (18)	−0.14491 (9)	0.0596 (7)
H10	0.617 (3)	1.0760 (16)	−0.1852 (9)	0.069 (5)*
C11	0.7926 (3)	0.97388 (18)	−0.10261 (9)	0.0619 (7)
H11	0.922 (3)	1.0175 (17)	−0.1118 (9)	0.072 (5)*
C12	0.7884 (3)	0.87486 (17)	−0.04569 (9)	0.0521 (6)
H12	0.917 (3)	0.8530 (15)	−0.0176 (9)	0.067 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0656 (9)	0.0566 (6)	0.0511 (6)	−0.0020 (5)	−0.0023 (5)	0.0048 (5)
O2	0.0418 (7)	0.0557 (6)	0.0551 (6)	0.0011 (5)	0.0037 (5)	0.0127 (5)
O3	0.0408 (8)	0.0946 (8)	0.0751 (8)	0.0062 (6)	−0.0033 (6)	0.0227 (6)
N1	0.0521 (10)	0.0499 (7)	0.0518 (8)	0.0018 (6)	0.0026 (6)	0.0090 (6)
C1	0.0539 (11)	0.0431 (7)	0.0458 (9)	0.0023 (7)	0.0056 (7)	−0.0043 (6)
C2	0.0589 (13)	0.0498 (9)	0.0740 (11)	0.0015 (8)	0.0217 (10)	0.0002 (8)
C3	0.0903 (18)	0.0522 (9)	0.0718 (12)	−0.0055 (10)	0.0368 (12)	−0.0006 (9)
C4	0.1016 (19)	0.0539 (10)	0.0491 (11)	−0.0050 (11)	0.0081 (10)	0.0044 (8)
C5	0.0443 (11)	0.0464 (8)	0.0498 (9)	0.0024 (7)	0.0052 (7)	−0.0011 (7)
C6	0.0350 (10)	0.0488 (7)	0.0506 (9)	0.0034 (6)	0.0043 (7)	−0.0060 (7)
C7	0.0347 (9)	0.0416 (7)	0.0447 (8)	0.0012 (6)	0.0061 (6)	−0.0067 (6)
C8	0.0385 (11)	0.0556 (8)	0.0577 (10)	−0.0016 (7)	0.0067 (8)	0.0046 (8)
C9	0.0482 (13)	0.0682 (10)	0.0627 (11)	0.0040 (8)	0.0019 (9)	0.0134 (8)
C10	0.0586 (13)	0.0624 (10)	0.0592 (11)	−0.0018 (9)	0.0134 (9)	0.0103 (8)
C11	0.0516 (13)	0.0694 (10)	0.0668 (11)	−0.0138 (9)	0.0171 (9)	0.0000 (9)
C12	0.0373 (11)	0.0643 (9)	0.0544 (10)	−0.0023 (8)	0.0032 (8)	−0.0035 (8)

Geometric parameters (Å, º)

O1—C1	1.3688 (17)	C5—H5	0.970 (15)
O1—C4	1.367 (2)	C6—C7	1.4795 (19)
O2—N1	1.4441 (14)	C7—C8	1.383 (2)
O2—C6	1.3523 (19)	C7—C12	1.379 (2)
O3—C6	1.1945 (18)	C8—H8	0.927 (16)
N1—C5	1.270 (2)	C8—C9	1.378 (2)
C1—C2	1.347 (3)	C9—H9	0.944 (17)
C1—C5	1.433 (2)	C9—C10	1.372 (3)
C2—H2	0.957 (17)	C10—H10	0.950 (16)
C2—C3	1.408 (2)	C10—C11	1.368 (2)
C3—H3	0.936 (18)	C11—H11	0.943 (17)
C3—C4	1.324 (4)	C11—C12	1.374 (2)
C4—H4	0.952 (16)	C12—H12	0.941 (16)
C1—O1—C4	105.29 (14)	O3—C6—C7	125.11 (14)
N1—O2—C6	113.27 (10)	C6—C7—C8	122.99 (13)
O2—N1—C5	106.73 (11)	C6—C7—C12	117.92 (13)
O1—C1—C2	110.27 (13)	C8—C7—C12	119.09 (13)
O1—C1—C5	119.30 (14)	C7—C8—H8	118.1 (8)
C2—C1—C5	130.42 (15)	C7—C8—C9	120.00 (15)
C1—C2—H2	126.0 (10)	H8—C8—C9	121.9 (9)
C1—C2—C3	106.37 (18)	C8—C9—H9	119.4 (10)
H2—C2—C3	127.6 (10)	C8—C9—C10	120.31 (16)
C2—C3—H3	127.2 (10)	H9—C9—C10	120.2 (10)
C2—C3—C4	107.0 (2)	C9—C10—H10	121.1 (10)
H3—C3—C4	125.7 (10)	C9—C10—C11	119.92 (16)
O1—C4—C3	111.09 (16)	H10—C10—C11	118.9 (10)
O1—C4—H4	114.1 (11)	C10—C11—H11	120.3 (9)
C3—C4—H4	134.8 (11)	C10—C11—C12	120.21 (17)
N1—C5—C1	122.35 (14)	H11—C11—C12	119.5 (10)
N1—C5—H5	121.6 (9)	C7—C12—C11	120.48 (15)
C1—C5—H5	116.0 (9)	C7—C12—H12	121.7 (10)
O2—C6—O3	123.68 (13)	C11—C12—H12	117.8 (10)
O2—C6—C7	111.21 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2	0.93 (2)	2.384 (13)	2.724 (2)	102 (1)
C5—H5···O3^{i}	0.97 (2)	2.312 (16)	3.159 (2)	145 (1)

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