research communications

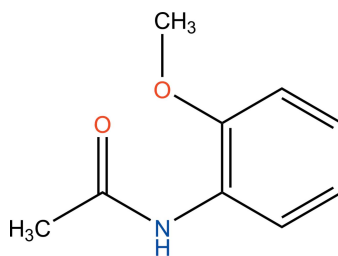## Hirshfeld surface analysis and crystal structure of N-(2-methoxyphenyl)acetamide

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The title compound, $C_9H_{11}NO_2$, was obtained as unexpected product from the reaction of (4-{2-benzyloxy-5-[(E)-(3-chloro-4-methylphenyl)diazenyl]benzyl-idene}-2-phenyloxazol-5(4H)-one) with 2-methoxyaniline in the presence of acetic acid as solvent. The amide group is not coplanar with the benzene ring, as shown by the C—N—C—O and C—N—C—C torsion angles of −2.5 (3) and 176.54 (19)°, respectively. Hirshfeld surface analysis and two-dimensional fingerprint plots indicate that the most important contributions to the crystal packing are from H···H (53.9%), C···H/H···C (21.4%), O···H/H···O (21.4%) and N···H/H···N (1.7%) interactions.

1. Chemical context

The amide function is one of the most important linkages in natural chemistry. It is the key linker in peptides and a number of polymers, and is additionally found in numerous pharmaceuticals and other items (Dam et al., 2010) with natural activity, including about 25% of commercially available drugs. Consequentially, the amide bond is a standout amongst the most vital changes in a current natural blend (Ojeda-Porras & Gamba-Sánchez, 2016). In the light of such discoveries, we report the crystal structure of the title compound.

2. Structural commentary

The molecular structure of the asymmetric unit of the $C_9H_{11}NO_2$ compound is shown in Fig. 1. The N1—C2, C2—O2 and C2—C1 bond lengths are 1.347 (2), 1.2285 (19) and 1.480 (3) Å, respectively. The C2—O2 bond in the amide group shows partial double-bond character and is similar in length to those found in amide compounds in the literature [1.215 (2) Å (Kansiz et al., 2018), 1.240 (2) Å (Aydemir et al., 2018) and 1.2205 (10) Å (Chkirate et al., 2019)]. The C3—C8
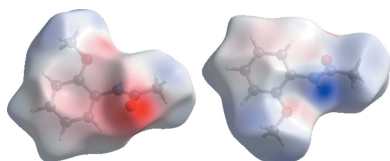
Table 1
Hydrogen-bond geometry (Å, °).

$Cg1$ is the centroid of the C3–C8 ring.

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
N1—H1$\cdots$O2^i	0.86	2.10	2.9486 (17)	168
C1—H1$B\cdots$O2^i	0.96	2.56	3.378 (2)	143
C1—H9$B\cdots$Cg1^{ii}	0.96	2.61	3.387	139

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

benzene ring is planar with an r.m.s. deviation of 0.0019. The amide group is not coplanar with the benzene ring, as shown by the C3—N1—C2—O2 and C3—N1—C2—C1 torsion angles of −2.5 (3) and 176.54 (19)°, respectively.

3. Supramolecular features

In the crystal, adjacent molecules are linked by weak C—H$\cdots$O hydrogen bonds, forming supramolecular chains
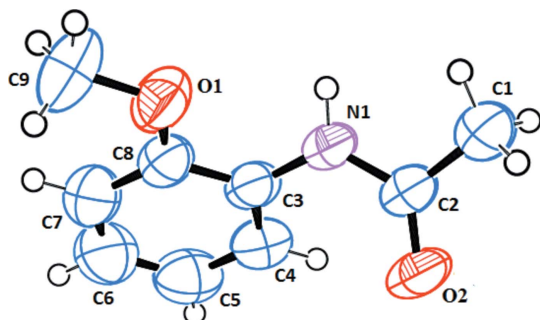

Figure 1
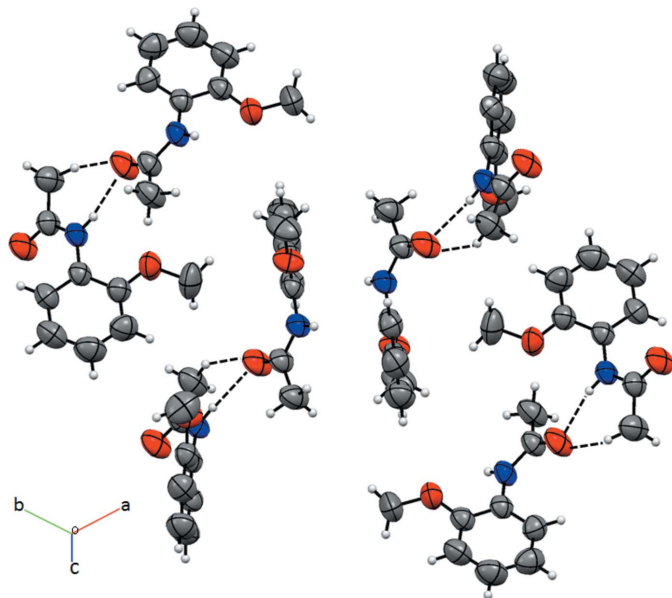The asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level.

Figure 2
A partial view of the crystal packing. Dashed lines denote the intermolecular C—H$\cdots$O and N—H$\cdots$O hydrogen bonds (Table 1).
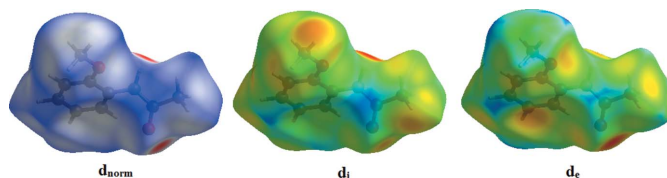

Figure 3
The Hirshfeld surface of the title compound mapped over d_{norm}, d_i and d_e.

propagating along the a-axis direction (Table 1 and Fig. 2). The chains are further connected by weak C—H$\cdots\pi$ interactions.

4. Hirshfeld surface analysis

Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were generated using *CrystalExplorer17* (Turner *et al.*, 2017). Plots of the Hirshfeld surface mapped over d_{norm}, d_i and d_e using a fixed colour scale of −0.5051 (red) to 1.2978 (blue) a.u. are shown in Fig. 3.. The red spots in the d_{norm} plot indicate the intermolecular contacts associated with the strong hydrogen bonds and interatomic contacts such as N—H$\cdots$O. Fig. 4 shows the d_{norm} mapped on the Hirshfeld surface to visualize the intermolecular interactions of the title compound. The fingerprint plots complement the Hirshfeld surface, quantitatively summarizing the nature and type of the intermolecular contacts by illustrating atom$_{inside}$/atom$_{outside}$ interactions (Fig. 5). The contribution from the H$\cdots$H
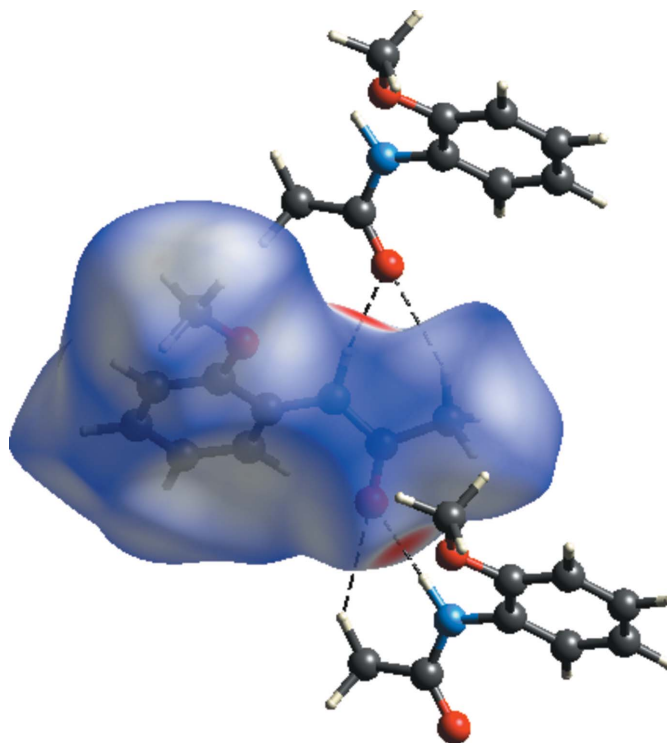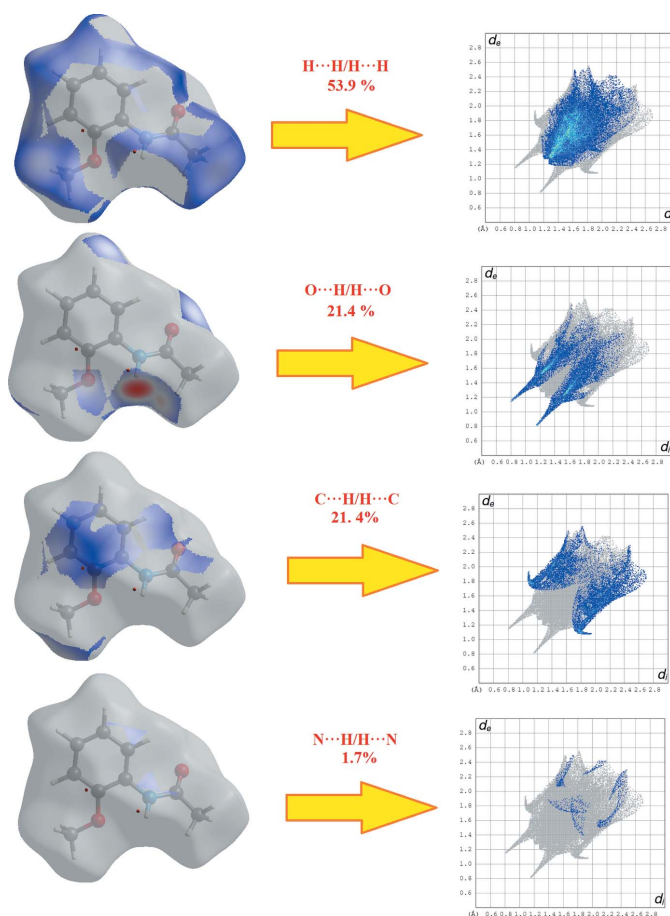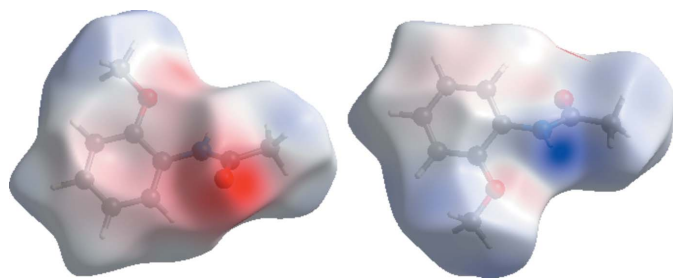

Figure 4
d_{norm} mapped on the Hirshfeld surface for visualizing the intermolecular interactions of the title compound.

Figure 5
Two-dimensional fingerprint plots with a d_{norm} view of the H···H/H···H (53.9%), C···H/H···C (21.4%), O···H/H···O (21.4%) and N···H/H···N (1.7%) contacts in the title compound.

contacts is observed to be highest towards the Hirshfeld surface with a 53.9% contribution. The contribution from the C—H···O hydrogen bond (21.4% contribution) appears as a pair of sharp spikes at $d_e + d_i$ =1.9 Å. A view of the three-dimensional Hirshfeld surface plotted over electrostatic potentials in the range −0.1028 to 0.1158 a.u. is shown in Fig. 6. The hydrogen-bond donors and acceptors are showed as blue and red regions around the atoms corresponding to positive and negative potentials, respectively.

Figure 6
The view of the three-dimensional Hirshfeld surface of the title compound plotted over the electrostatic potentials.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_{11}NO_2$
M_r	165.19
Crystal system, space group	Orthorhombic, $Pbca$
Temperature (K)	296
a, b, c (Å)	9.5115 (7), 18.7385 (19), 10.0216 (8)
V (Å^3)	1786.2 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	$0.43 \times 0.39 \times 0.37$
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (X-$RED32$; Stoe & Cie, 2002)
T_{min}, T_{max}	0.946, 0.978
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14575, 1748, 1168
R_{int}	0.090
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.148, 1.05
No. of reflections	1748
No. of parameters	111
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.13, −0.12

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT2018* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae et al., 2006) and *PLATON* (Spek, 2009).

5. Database survey

A search in the Cambridge Structural Database (CSD version 5.39, update of August 2018; Groom et al., 2016) for *N*-(2-methoxyphenyl)acetamide derivatives found several similar structures: 3-hydroxy-7,8-dimethoxyquinolin-2(1*H*)-one (BIZGAT; Song et al., 2008), 1-(2-methoxyphenyl)-1*H*-pyrrole-2,5-dione (XEBZIP; Sirajuddin et al., 2012) and *cis*-cyclohexane-1,2-carboxylic anhydride with *o*- and *p*-anisidine and *m*- and *p*-aminobenzoic acids (BECVAI; Smith et al., 2012). In the structure of BIZGAT, the molecules are linked into chains by N—H···O hydrogen bonds as in the title structure.

6. Synthesis and crystallization

This compound was formed as by-product in the synthesis of a benzamide derivative from the reaction between an oxazolone with *o*- methoxyaniline (Samad & Hawaiz, 2019) in the presence of acetic acid as solvent. The reaction mixture was refluxed for 2 h, cooled, poured into water, filtered and dried. The remaining filtrate was left for seven days to obtain good-quality crystals.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were positioned geometrically and refined using a riding model with C—H =

0.93 Å for aromatic H atoms, C—H = 0.96 Å for methyl H atoms, and with $U_{iso}(H) = 1.2–1.5\ U_{eq}(C)$.

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supporting information

Hirshfeld surface analysis and crystal structure of *N*-(2-methoxyphenyl)-acetamide

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2018* (Sheldrick, 2015b), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

N-(2-Methoxyphenyl)acetamide

Crystal data

$C_9H_{11}NO_2$	$D_x = 1.229$ Mg m^{-3}
$M_r = 165.19$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 26458 reflections
$a = 9.5115$ (7) Å	$\theta = 2.0$–28.3°
$b = 18.7385$ (19) Å	$\mu = 0.09$ mm^{-1}
$c = 10.0216$ (8) Å	$T = 296$ K
$V = 1786.2$ (3) Å^3	Prism, yellow
$Z = 8$	$0.43 \times 0.39 \times 0.37$ mm
$F(000) = 704$	

Data collection

Stoe IPDS 2 diffractometer	$T_{min} = 0.946$, $T_{max} = 0.978$
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	14575 measured reflections
	1748 independent reflections
Plane graphite monochromator	1168 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.090$
rotation method scans	$\theta_{max} = 26.0°$, $\theta_{min} = 2.2°$
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$h = -11 \rightarrow 10$
	$k = -22 \rightarrow 22$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	111 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.050$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.148$	
$S = 1.05$	H-atom parameters constrained
1748 reflections	

$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
O1	0.64879 (13)	0.70439 (8)	0.55583 (15)	0.0835 (5)
O2	0.25840 (10)	0.58665 (10)	0.69551 (15)	0.0943 (6)
N1	0.49079 (12)	0.60150 (8)	0.65857 (16)	0.0654 (5)
H1	0.572027	0.603270	0.696112	0.078*
C3	0.48608 (15)	0.61300 (10)	0.5196 (2)	0.0622 (5)
C8	0.57097 (16)	0.66649 (11)	0.4655 (2)	0.0678 (5)
C2	0.37959 (16)	0.58800 (10)	0.7378 (2)	0.0702 (5)
C4	0.40284 (18)	0.57277 (11)	0.4362 (2)	0.0739 (6)
H4	0.346101	0.537006	0.471444	0.089*
C7	0.5712 (2)	0.67836 (14)	0.3299 (2)	0.0872 (7)
H7	0.627687	0.713842	0.293428	0.105*
C1	0.4131 (2)	0.57364 (14)	0.8795 (2)	0.0937 (8)
H1A	0.349072	0.599462	0.935617	0.141*
H1B	0.507637	0.588706	0.897970	0.141*
H1C	0.404506	0.523450	0.896863	0.141*
C5	0.4032 (2)	0.58533 (13)	0.3000 (3)	0.0917 (7)
H5	0.346474	0.558364	0.243764	0.110*
C6	0.4874 (2)	0.63744 (16)	0.2492 (3)	0.0996 (8)
H6	0.487972	0.645447	0.157579	0.119*
C9	0.7372 (2)	0.75987 (14)	0.5065 (3)	0.1092 (9)
H9A	0.802856	0.740327	0.443554	0.164*
H9B	0.787667	0.781102	0.579383	0.164*
H9C	0.680779	0.795524	0.463403	0.164*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0703 (8)	0.0959 (10)	0.0843 (11)	−0.0264 (7)	0.0062 (7)	−0.0008 (8)
O2	0.0401 (6)	0.1531 (15)	0.0896 (11)	−0.0056 (7)	0.0017 (6)	0.0087 (10)
N1	0.0397 (6)	0.0882 (11)	0.0682 (11)	−0.0052 (6)	−0.0012 (6)	0.0051 (8)
C3	0.0445 (7)	0.0737 (11)	0.0683 (13)	0.0031 (7)	0.0007 (7)	−0.0008 (9)
C8	0.0530 (8)	0.0804 (12)	0.0699 (14)	0.0004 (9)	0.0043 (8)	0.0014 (10)
C2	0.0459 (8)	0.0903 (14)	0.0744 (14)	−0.0029 (9)	0.0031 (8)	0.0061 (11)
C4	0.0579 (9)	0.0824 (13)	0.0813 (16)	−0.0002 (9)	−0.0059 (9)	−0.0065 (11)
C7	0.0767 (13)	0.1117 (18)	0.0732 (17)	0.0005 (12)	0.0110 (11)	0.0104 (13)
C1	0.0611 (11)	0.142 (2)	0.0778 (16)	−0.0005 (12)	0.0054 (10)	0.0168 (14)

C5	0.0773 (12)	0.1154 (19)	0.0823 (17)	0.0060 (13)	−0.0135 (12)	−0.0179 (14)
C6	0.0955 (16)	0.134 (2)	0.0697 (16)	0.0068 (15)	0.0001 (12)	0.0016 (16)
C9	0.0960 (15)	0.1067 (18)	0.125 (2)	−0.0392 (14)	0.0343 (15)	−0.0085 (17)

Geometric parameters (Å, º)

O1—C8	1.368 (2)	C7—C6	1.370 (3)
O1—C9	1.426 (2)	C7—H7	0.9300
O2—C2	1.2285 (19)	C1—H1A	0.9600
N1—C2	1.347 (2)	C1—H1B	0.9600
N1—C3	1.410 (2)	C1—H1C	0.9600
N1—H1	0.8600	C5—C6	1.362 (3)
C3—C4	1.376 (3)	C5—H5	0.9300
C3—C8	1.397 (3)	C6—H6	0.9300
C8—C7	1.377 (3)	C9—H9A	0.9600
C2—C1	1.480 (3)	C9—H9B	0.9600
C4—C5	1.385 (3)	C9—H9C	0.9600
C4—H4	0.9300		
C8—O1—C9	117.93 (18)	C2—C1—H1A	109.5
C2—N1—C3	125.90 (14)	C2—C1—H1B	109.5
C2—N1—H1	117.1	H1A—C1—H1B	109.5
C3—N1—H1	117.1	C2—C1—H1C	109.5
C4—C3—C8	119.4 (2)	H1A—C1—H1C	109.5
C4—C3—N1	122.29 (17)	H1B—C1—H1C	109.5
C8—C3—N1	118.33 (16)	C6—C5—C4	119.5 (2)
O1—C8—C7	124.65 (18)	C6—C5—H5	120.2
O1—C8—C3	115.38 (18)	C4—C5—H5	120.2
C7—C8—C3	119.97 (19)	C5—C6—C7	121.5 (2)
O2—C2—N1	122.48 (19)	C5—C6—H6	119.3
O2—C2—C1	122.00 (16)	C7—C6—H6	119.3
N1—C2—C1	115.51 (15)	O1—C9—H9A	109.5
C3—C4—C5	120.2 (2)	O1—C9—H9B	109.5
C3—C4—H4	119.9	H9A—C9—H9B	109.5
C5—C4—H4	119.9	O1—C9—H9C	109.5
C6—C7—C8	119.5 (2)	H9A—C9—H9C	109.5
C6—C7—H7	120.3	H9B—C9—H9C	109.5
C8—C7—H7	120.3		
C2—N1—C3—C4	−41.9 (3)	C3—N1—C2—C1	176.54 (19)
C2—N1—C3—C8	139.18 (19)	C8—C3—C4—C5	−0.1 (3)
C9—O1—C8—C7	−0.4 (3)	N1—C3—C4—C5	−179.04 (17)
C9—O1—C8—C3	−179.83 (17)	O1—C8—C7—C6	−179.23 (19)
C4—C3—C8—O1	179.21 (15)	C3—C8—C7—C6	0.2 (3)
N1—C3—C8—O1	−1.8 (2)	C3—C4—C5—C6	0.5 (3)
C4—C3—C8—C7	−0.2 (3)	C4—C5—C6—C7	−0.6 (3)
N1—C3—C8—C7	178.77 (17)	C8—C7—C6—C5	0.2 (4)
C3—N1—C2—O2	−2.5 (3)		

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the C3–C8 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2ⁱ	0.86	2.10	2.9486 (17)	168
C1—H1*B*···O2ⁱ	0.96	2.56	3.378 (2)	143
C1—H9*B*···*Cg*1ⁱⁱ	0.96	2.61	3.387	139

Symmetry codes: (i) *x*+1/2, *y*, −*z*+3/2; (ii) −*x*−1, *y*+1/2, −*z*+3/2.