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Structural study and Hirshfeld surface analysis of (*Z*)-4-(2-methoxybenzylidene)-3-phenylisoxazol-5(4*H*)-one

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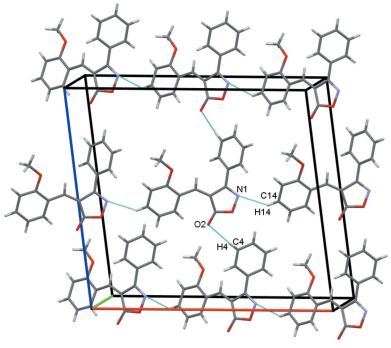
The title compound, $C_{17}H_{13}NO_3$, adopts a *Z* configuration about the C=C bond. The isoxazole and methoxybenzylidene rings are almost coplanar with a dihedral angle of 9.63 (7)° between them. In contrast, the phenyl substituent is twisted significantly out of the plane of the oxazole ring, with the two rings inclined to each other by 46.22 (4)°. The crystal structure features C—H···O, C—H···N and C—H···π hydrogen bonds and π–π contacts. An analysis of the Hirshfeld surfaces points to the importance of H···H, H···C/C···H and H···O/O···H contacts. The included surface areas of the title compound were compared to those of the isomeric structure (*Z*)-4-(4-methoxybenzylidene)-3-phenylisoxazol-5(4*H*)-one [Zhang *et al.* (2015). *CrystEngComm*, **17**, 7316–7322].

1. Chemical context

Isoxazolones are known to be inhibitors of the factorization of tumor necrosis alpha (TNF-α) (Laughlin *et al.*, 2005), antimicrobial agents (Mazimba *et al.*, 2014), as drugs for the treatment of cerebrovascular disorders and as muscle relaxants. In agriculture, they are used as herbicides (Guo, *et al.*, 2020) and fungicides (Miyake *et al.*, 2012). They undergo various chemical transformations (Batra *et al.*, 1994) and are excellent intermediates in the synthesis of various heterocycles, including pyridopyrimidines (Tu *et al.*, 2006), quinolines (Abbiati *et al.*, 2003) and polycycles (Badrey & Gomha, 2014). Because of their importance, these compounds have been studied extensively and several procedures for their synthesis are described using a three-component polycondensation between an aromatic aldehyde, ethyl acetoacetate and hydroxylamine hydrochloride under different conditions (Liu *et al.*, 2011; Fozooni *et al.*, 2013).



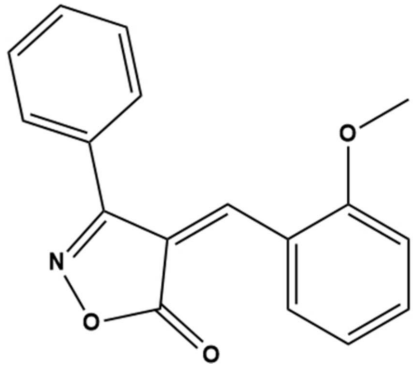


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Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.53	3.463 (2)	176
C5—H5···O2^{ii}	0.93	2.81	3.728 (2)	169
C10—H10···O3	0.93	2.26	2.7009 (18)	108
C12—H12···O2	0.93	2.15	2.998 (2)	151
C14—H14···N1^{iii}	0.93	2.58	3.396 (2)	147
C17—H17*A*···O3^{iv}	0.96	2.78	3.615 (2)	147
C17—H17*C*···*Cg*^{iv}	0.96	2.82	3.606 (2)	139

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

We report here on the use of K_2CO_3 as very inexpensive, highly available and safe catalyst in an organic medium for isoxazolone formation and we describe the synthesis, molecular and crystal structures, and Hirshfeld surface analysis of the title isoxazole derivative, **1** (Fig. 1).

2. Structural commentary

The asymmetric unit contains one molecule and the molecule adopts a *Z* configuration about the C8═C10 bond. The entire (*Z*)-4-(2-methoxybenzylideneisoxazolone) segment of the molecule is almost planar with an r.m.s. deviation from the mean plane through all 15 non-hydrogen atoms of the fragment of only 0.0927 Å. This conformation is supported by the formation of an intramolecular C12—H12···O1 hydrogen bond (Table 1), which links the isoxazole ring and the benzene ring of the 2-methoxybenzylidene substituent. These two rings are inclined to one another at an angle of 9.63 (7)°. The (C1–C6) phenyl substituent is twisted out of this plane, the phenyl and isoxazole rings being inclined to one another by 46.22 (4)°. Bond lengths and angles agree well with those found in the isomeric derivative **2** (Zhang *et al.*, 2015) and also with the values observed for the related compound (4*Z*)-4-benzylidene-2-phenyl-1,3-oxazol5(4*H*)-one (Asiri *et al.*, 2012).

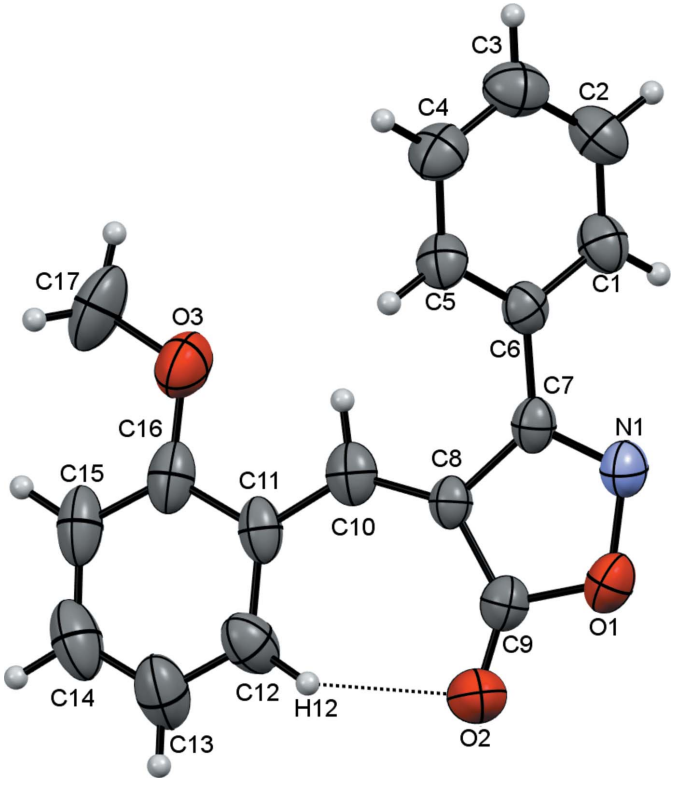

Figure 1
The molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is shown as a black dashed line.

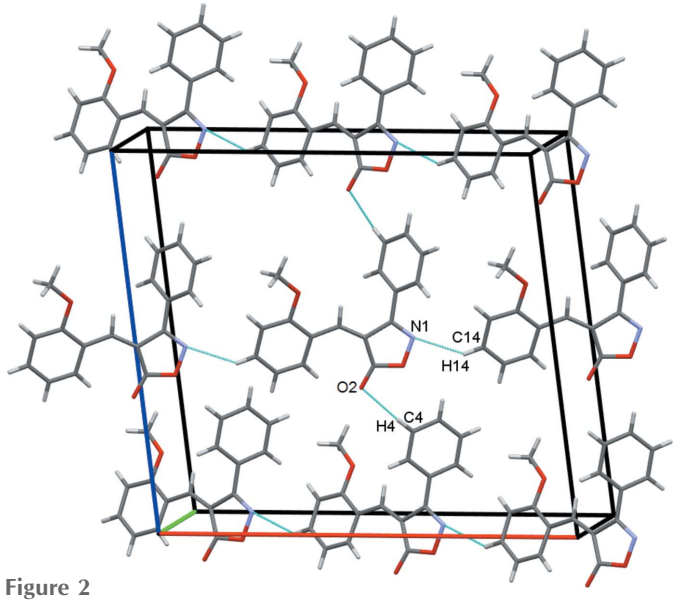

Figure 2
Sheets of molecules of **1** in the *ac* plane.

3. Supramolecular features

In the crystal, molecules stack along the *b*-axis direction (Fig. 2). Molecules are connected by C4—H4···O1^{i} and C14—

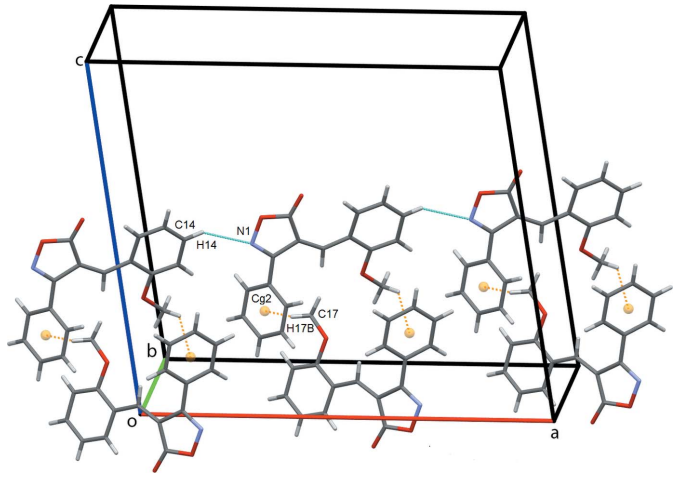

Figure 3
Double rows of molecules of **1** along the *a*-axis direction. *Cg*2 is the centroid of the C1–C6 phenyl ring, shown here as an orange sphere, with the C—H···π contacts drawn as orange dashed lines.

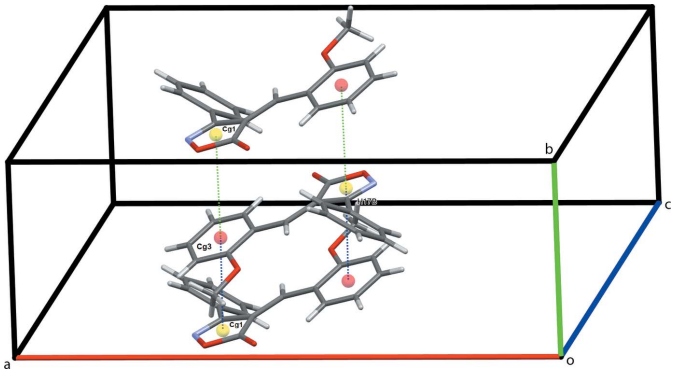

Figure 4
π–π contacts for 1 stacking molecules along the *b*-axis direction. *Cg*1 and *Cg*3 are the centroids of the N1/O2/C7–C9 isoxazole and the C11–C16 benzene rings, respectively. The two discrete π–π contacts *Cg*1$\cdots$*Cg*3 = 3.7049 (9) and 3.9200 (9) Å are shown as green and blue dashed lines, respectively.

H14$\cdots$N1^{iii} hydrogen bonds, leading to the formation of sheets in the *ac* plane, Fig. 3. C—H$\cdots\pi$ contacts between the methoxymethyl group and the C1–C6 phenyl ring form double chains of molecules along the *a*-axis direction, supported by the above-mentioned C14—H14$\cdots$N1^{iii} hydrogen bonds, Fig. 4. Intermolecular H$\cdots$O short contacts are also present [C17—H17*A*$\cdots$O3^{iv} = 2.78 Å and C5—H5$\cdots$O2^{ii} = 2.81 Å]. Two π–π contacts [3.7049 (9) and 3.9200 (9) Å] are found between the centroids of the isoxazolone ring and the methoxy-substituted benzene ring, which stack adjacent molecules in an obverse fashion along *b*.

4. Analysis of the Hirshfeld surfaces

Further details of the intermolecular interactions in **1** were obtained using Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) with Hirshfeld surfaces and two-dimensional fingerprint plots (McKinnon *et al.*, 2007) generated using *CrystalExplorer* (Turner *et al.* 2017). Fig. 5 shows the Hirshfeld surfaces for opposite faces of the asymmetric unit of molecule **1**. The bright red circles correspond to C—H$\cdots$N and C—H$\cdots$O hydrogen bonds while a weaker C—H$\cdots\pi$ contact appears as a faint red circle. Fingerprint plots for **1** are shown in Fig. 6. As the CIF file for the isomeric molecule, **2**, was available from the CCD, it was of interest to compare and contrast contributions to the included surface areas from the two isomers as shown in Table 2. As expected, H$\cdots$H contacts are the most prolific in both cases. Other contributions were generally very similar, the sole exception being that the C$\cdots$O/O$\cdots$C contacts made up almost twice the surface area for **2** as for **1**. The change from the 2- to the 4-position in **2** may allow the methoxy substituent in **2** to contribute more substantially to the surface of the molecule.

5. Database survey

A search of the Cambridge Structural Database (CSD, V3.59, last update February 2019; Groom *et al.*, 2016) for (*Z*)-4-benzylidene-3-phenylsoxazol-5(4*H*)-one yielded seventeen hits. Importantly, one of these, *i.e.* (*Z*)-4-(4-methoxybenzylidene)-3-phenylisoxazol-5(4*H*)-one (SULZAC; Zhang *et al.*, 2015) is an isomer (**2**) of the title compound with the methoxy substituent in the 4-position of the benzene ring. Another paper (Jiang *et al.*, 2013) included the closely related compound (*Z*)-4-(4-[dimethylamino)benzylidene]-3-phenylisoxazol-5(4*H*)-one (IDIBEE) together with two other related compounds, IDIBII and IDIBOO, that exhibit large second harmonic generation effects. The search also revealed four other structures in which the configuration about the C=C

Table 2
Short contacts and contributions (%) to the Hirshfeld surface for **1** and **2**.

Contact	**1**	**2**
H$\cdots$H	40.8	40.5
H$\cdots$C/C$\cdots$H	19.4	18.1
H$\cdots$O/O$\cdots$H	19.7	19.6
H$\cdots$N/N$\cdots$H	6.4	5.3
C$\cdots$C	7.9	6.5
C$\cdots$O/O$\cdots$C	3.6	6.9
C$\cdots$N/N$\cdots$C	1.8	2.9
O$\cdots$O	0.6	0.1
N$\cdots$N	0.1	

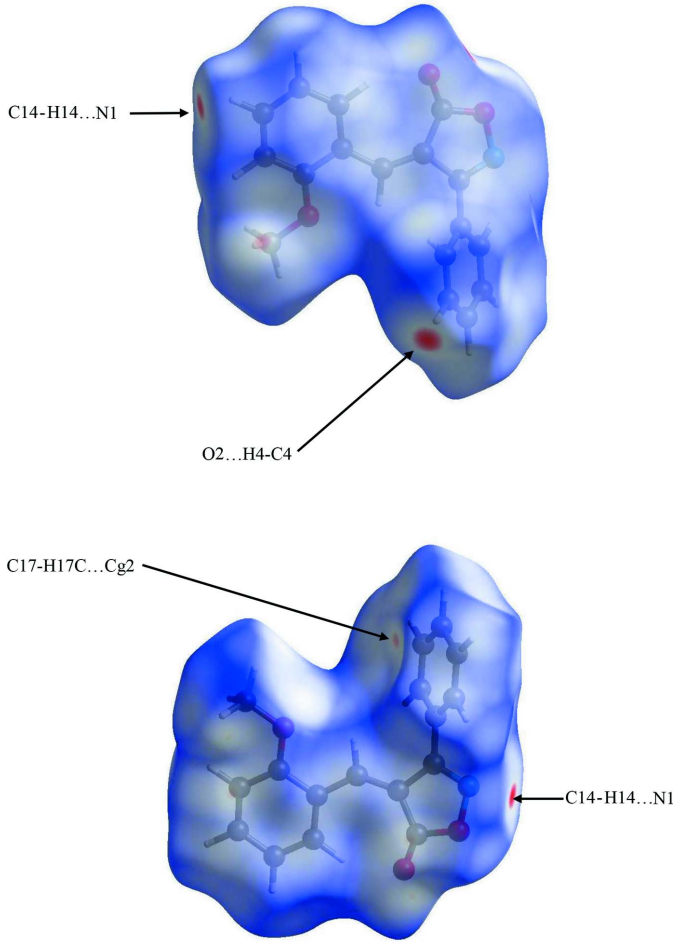

Figure 5
Hirshfeld surfaces for opposite faces of the molecule of **1**, mapped over d_{norm} in the range −0.1701 to 1.4088 a.u.

bond is *Z*, namely 4-(2-hydroxybenzylidene)-3-methylisoxazol-5(4*H*)-one (AJESAK; Cheng *et al.*, 2009), (4*Z*)-4-benzylidene-2-phenyl-1,3-oxazol-5(4*H*)-one (YAXMUH; Asiri *et al.*, 2012), (*Z*)-4-benzylidene-3-methylisoxazol-5(4*H*)-one [MBYIOZ (Meunier-Piret *et al.*, 1972) and MBYIOZ01 (Chandra *et al.*, 2012)] and a recent addition, (*Z*)-4-(4-hydroxybenzylidene)-3-methylisoxazol-5(4*H*)-one (Zemamouche *et al.*, 2018).

6. Synthesis and crystallization

2-Methoxybenzaldehyde (1 mmol), hydroxyamine hydrochloride (1 mmol), ethyl benzoylacetate (1 mmol) and K_2CO_3 (5 mol%) were mixed in a 25 ml flask equipped with a magnetic stirrer. The mixture was refluxed in 5 ml of water for 2.5 h (the reaction was monitored by TLC). On completion of the reaction, the mixture was gradually poured into ice-cold water. Stirring was maintained for a few minutes and the resulting solid was filtered and purified by crystallization from ethanol.

7. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C-methyl).

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}NO_3$
M_r	279.28
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	20.3883 (6), 7.5925 (2), 17.9858 (5)
β (°)	95.791 (1)
V (Å^3)	2769.96 (13)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.32 × 0.23 × 0.10
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.98, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	35544, 2495, 1548
R_{int}	0.12
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.600
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.101, 1.00
No. of reflections	2495
No. of parameters	190
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.09, −0.16

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SIR92* (Altomare *et al.*, 1994), *SHELXL2018/3* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2020) and *WinGX* publication routines (Farrugia, 2012).

Acknowledgements

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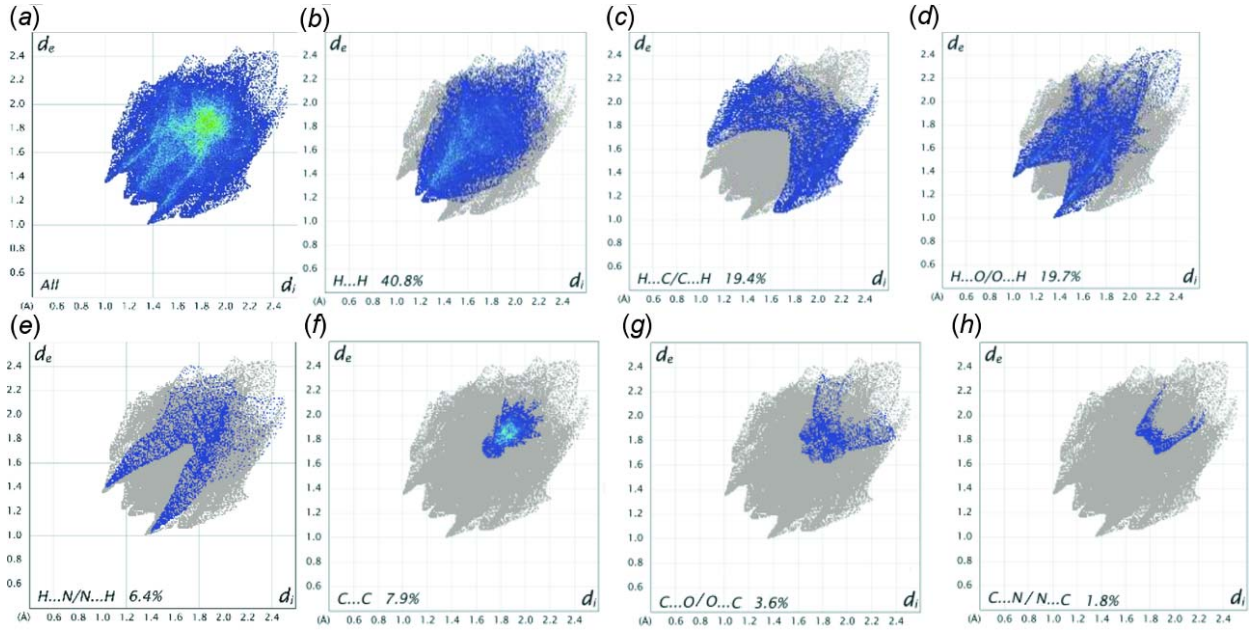

Figure 6
(*a*) The two-dimensional fingerprint plot for all interactions, together with those (*b*)–(*h*) delineated into individual contact types with included surface areas for the major individual contacts. Minor contacts contributing less than 1% to the total surface area are not shown here but, for completeness, are included in Table 2.

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supporting information

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Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *APEX2* (Bruker, 2009); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012).

(*Z*)-4-(2-Methoxybenzylidene)-3-phenylisoxazol-5(4*H*)-one

Crystal data

$C_{17}H_{13}NO_3$
$M_r = 279.28$
Monoclinic, $C2/c$
$a = 20.3883$ (6) Å
$b = 7.5925$ (2) Å
$c = 17.9858$ (5) Å
$\beta = 95.791$ (1)°
$V = 2769.96$ (13) Å^3
$Z = 8$
$F(000) = 1168$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1218 reflections
$\theta = 2.3$–33.4°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Needle, white
0.32 × 0.23 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
$T_{min} = 0.98$, $T_{max} = 0.99$
35544 measured reflections
2495 independent reflections
1548 reflections with $I > 2\sigma(I)$
$R_{int} = 0.12$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.3°$
$h = -24\rightarrow24$
$k = -9\rightarrow9$
$l = -21\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.101$
$S = 1.00$
2495 reflections
190 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.09$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ 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	0.63879 (5)	0.40128 (15)	0.44786 (5)	0.0604 (3)
O2	0.53785 (6)	0.34750 (15)	0.39356 (6)	0.0641 (4)
O3	0.44474 (6)	0.07225 (16)	0.65984 (6)	0.0672 (4)
N1	0.67268 (6)	0.40557 (18)	0.52191 (7)	0.0559 (4)
C11	0.45217 (7)	0.17739 (18)	0.53778 (8)	0.0423 (4)
C10	0.51760 (7)	0.23684 (17)	0.56343 (8)	0.0409 (4)
H10	0.528401	0.219863	0.614403	0.049*
C6	0.65205 (7)	0.35594 (18)	0.64799 (8)	0.0419 (4)
C5	0.61267 (8)	0.43223 (19)	0.69787 (8)	0.0499 (4)
H5	0.571966	0.479668	0.680373	0.060*
C8	0.56690 (7)	0.31231 (18)	0.52928 (7)	0.0376 (4)
C7	0.63081 (7)	0.35667 (18)	0.56711 (8)	0.0400 (4)
C12	0.42284 (8)	0.1993 (2)	0.46438 (9)	0.0566 (5)
H12	0.446785	0.253003	0.429297	0.068*
C1	0.71264 (8)	0.2846 (2)	0.67501 (9)	0.0518 (4)
H1	0.739346	0.232764	0.642170	0.062*
C16	0.41449 (8)	0.0947 (2)	0.58918 (9)	0.0511 (4)
C3	0.69408 (9)	0.3668 (2)	0.79957 (9)	0.0633 (5)
H3	0.708286	0.370488	0.850331	0.076*
C9	0.57442 (8)	0.3509 (2)	0.45076 (8)	0.0474 (4)
C2	0.73315 (9)	0.2905 (2)	0.75030 (10)	0.0632 (5)
H2	0.773723	0.242656	0.768033	0.076*
C4	0.63365 (9)	0.4380 (2)	0.77336 (9)	0.0593 (5)
H4	0.607189	0.489740	0.806480	0.071*
C15	0.35023 (8)	0.0404 (2)	0.56730 (11)	0.0633 (5)
H15	0.325381	−0.013537	0.601461	0.076*
C14	0.32368 (9)	0.0672 (2)	0.49456 (12)	0.0717 (6)
H14	0.280483	0.032695	0.480253	0.086*
C13	0.35978 (9)	0.1436 (2)	0.44307 (11)	0.0710 (6)
H13	0.341624	0.157705	0.393888	0.085*
C17	0.41045 (9)	−0.0215 (3)	0.71332 (9)	0.0764 (6)
H17A	0.437527	−0.026963	0.760129	0.115*
H17B	0.370029	0.038399	0.720143	0.115*
H17C	0.400829	−0.138807	0.695475	0.115*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0531 (8)	0.0810 (9)	0.0494 (6)	−0.0098 (6)	0.0165 (5)	0.0092 (5)
O2	0.0606 (8)	0.0864 (9)	0.0452 (6)	−0.0005 (6)	0.0045 (5)	0.0073 (5)
O3	0.0630 (8)	0.0842 (9)	0.0568 (7)	−0.0219 (6)	0.0176 (6)	0.0039 (6)
N1	0.0455 (9)	0.0667 (9)	0.0564 (8)	−0.0096 (7)	0.0101 (7)	0.0045 (7)
C11	0.0345 (9)	0.0357 (8)	0.0577 (9)	−0.0008 (7)	0.0086 (7)	−0.0027 (7)
C10	0.0416 (9)	0.0378 (9)	0.0437 (7)	0.0012 (7)	0.0055 (7)	−0.0010 (6)
C6	0.0362 (9)	0.0402 (9)	0.0494 (8)	−0.0053 (7)	0.0054 (7)	−0.0032 (7)
C5	0.0447 (10)	0.0503 (10)	0.0545 (9)	−0.0012 (8)	0.0036 (8)	−0.0042 (7)
C8	0.0354 (9)	0.0362 (8)	0.0421 (7)	−0.0012 (7)	0.0083 (6)	0.0011 (6)
C7	0.0353 (9)	0.0371 (9)	0.0490 (8)	−0.0024 (7)	0.0103 (7)	0.0008 (6)
C12	0.0477 (11)	0.0553 (11)	0.0653 (10)	−0.0036 (8)	−0.0009 (8)	0.0075 (8)
C1	0.0401 (10)	0.0558 (10)	0.0593 (9)	−0.0014 (8)	0.0042 (8)	−0.0076 (8)
C16	0.0454 (11)	0.0462 (10)	0.0639 (10)	−0.0059 (8)	0.0161 (8)	−0.0066 (8)
C3	0.0719 (14)	0.0657 (12)	0.0499 (9)	−0.0098 (10)	−0.0056 (9)	−0.0071 (8)
C9	0.0471 (11)	0.0485 (10)	0.0478 (8)	−0.0001 (8)	0.0109 (8)	0.0042 (7)
C2	0.0525 (11)	0.0653 (12)	0.0680 (11)	−0.0018 (9)	−0.0123 (10)	−0.0009 (9)
C4	0.0619 (13)	0.0622 (12)	0.0547 (9)	−0.0024 (9)	0.0112 (9)	−0.0136 (8)
C15	0.0442 (12)	0.0619 (12)	0.0865 (13)	−0.0126 (9)	0.0204 (9)	−0.0081 (10)
C14	0.0387 (11)	0.0725 (13)	0.1023 (15)	−0.0101 (9)	−0.0008 (11)	−0.0090 (11)
C13	0.0491 (12)	0.0754 (14)	0.0847 (13)	−0.0095 (10)	−0.0118 (10)	0.0106 (10)
C17	0.0838 (14)	0.0802 (14)	0.0719 (11)	−0.0161 (11)	0.0400 (10)	0.0042 (10)

Geometric parameters (Å, º)

O1—C9	1.3729 (18)	C12—C13	1.371 (2)
O1—N1	1.4376 (15)	C12—H12	0.9300
O2—C9	1.2085 (17)	C1—C2	1.377 (2)
O3—C16	1.3663 (18)	C1—H1	0.9300
O3—C17	1.4341 (18)	C16—C15	1.392 (2)
N1—C7	1.2914 (18)	C3—C2	1.378 (2)
C11—C12	1.403 (2)	C3—C4	1.384 (2)
C11—C16	1.408 (2)	C3—H3	0.9300
C11—C10	1.4394 (19)	C2—H2	0.9300
C10—C8	1.3566 (19)	C4—H4	0.9300
C10—H10	0.9300	C15—C14	1.379 (2)
C6—C5	1.389 (2)	C15—H15	0.9300
C6—C1	1.391 (2)	C14—C13	1.369 (2)
C6—C7	1.4754 (19)	C14—H14	0.9300
C5—C4	1.3824 (19)	C13—H13	0.9300
C5—H5	0.9300	C17—H17A	0.9600
C8—C7	1.4475 (19)	C17—H17B	0.9600
C8—C9	1.4654 (18)	C17—H17C	0.9600
C9—O1—N1	110.05 (10)	C15—C16—C11	120.45 (16)
C16—O3—C17	118.75 (13)	C2—C3—C4	119.86 (15)

C7—N1—O1	106.87 (12)	C2—C3—H3	120.1
C12—C11—C16	117.50 (15)	C4—C3—H3	120.1
C12—C11—C10	123.89 (14)	O2—C9—O1	118.92 (13)
C16—C11—C10	118.61 (14)	O2—C9—C8	134.47 (15)
C8—C10—C11	133.82 (13)	O1—C9—C8	106.61 (12)
C8—C10—H10	113.1	C1—C2—C3	120.56 (16)
C11—C10—H10	113.1	C1—C2—H2	119.7
C5—C6—C1	119.18 (14)	C3—C2—H2	119.7
C5—C6—C7	120.26 (14)	C5—C4—C3	119.91 (16)
C1—C6—C7	120.52 (14)	C5—C4—H4	120.0
C4—C5—C6	120.39 (15)	C3—C4—H4	120.0
C4—C5—H5	119.8	C14—C15—C16	119.51 (17)
C6—C5—H5	119.8	C14—C15—H15	120.2
C10—C8—C7	123.95 (12)	C16—C15—H15	120.2
C10—C8—C9	132.47 (13)	C13—C14—C15	121.19 (17)
C7—C8—C9	103.27 (12)	C13—C14—H14	119.4
N1—C7—C8	113.07 (13)	C15—C14—H14	119.4
N1—C7—C6	118.35 (13)	C14—C13—C12	119.65 (17)
C8—C7—C6	128.56 (13)	C14—C13—H13	120.2
C13—C12—C11	121.68 (17)	C12—C13—H13	120.2
C13—C12—H12	119.2	O3—C17—H17A	109.5
C11—C12—H12	119.2	O3—C17—H17B	109.5
C2—C1—C6	120.10 (16)	H17A—C17—H17B	109.5
C2—C1—H1	119.9	O3—C17—H17C	109.5
C6—C1—H1	119.9	H17A—C17—H17C	109.5
O3—C16—C15	123.35 (16)	H17B—C17—H17C	109.5
O3—C16—C11	116.20 (14)		

C9—O1—N1—C7	1.14 (16)	C17—O3—C16—C15	4.0 (2)
C12—C11—C10—C8	−4.4 (3)	C17—O3—C16—C11	−175.92 (14)
C16—C11—C10—C8	176.65 (15)	C12—C11—C16—O3	178.84 (14)
C1—C6—C5—C4	0.4 (2)	C10—C11—C16—O3	−2.2 (2)
C7—C6—C5—C4	−177.39 (13)	C12—C11—C16—C15	−1.1 (2)
C11—C10—C8—C7	−177.90 (14)	C10—C11—C16—C15	177.91 (14)
C11—C10—C8—C9	−5.5 (3)	N1—O1—C9—O2	176.89 (13)
O1—N1—C7—C8	1.18 (17)	N1—O1—C9—C8	−2.88 (15)
O1—N1—C7—C6	−177.58 (11)	C10—C8—C9—O2	10.1 (3)
C10—C8—C7—N1	171.42 (14)	C7—C8—C9—O2	−176.37 (17)
C9—C8—C7—N1	−2.85 (17)	C10—C8—C9—O1	−170.22 (15)
C10—C8—C7—C6	−10.0 (2)	C7—C8—C9—O1	3.35 (15)
C9—C8—C7—C6	175.75 (14)	C6—C1—C2—C3	0.0 (2)
C5—C6—C7—N1	132.05 (16)	C4—C3—C2—C1	0.1 (2)
C1—C6—C7—N1	−45.7 (2)	C6—C5—C4—C3	−0.3 (2)
C5—C6—C7—C8	−46.5 (2)	C2—C3—C4—C5	0.1 (2)
C1—C6—C7—C8	135.77 (16)	O3—C16—C15—C14	−179.53 (15)
C16—C11—C12—C13	0.3 (2)	C11—C16—C15—C14	0.4 (2)
C10—C11—C12—C13	−178.59 (15)	C16—C15—C14—C13	1.1 (3)
C5—C6—C1—C2	−0.2 (2)	C15—C14—C13—C12	−1.9 (3)

C7—C6—C1—C2	177.52 (14)	C11—C12—C13—C14	1.1 (3)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.53	3.463 (2)	176
C5—H5···O2^{ii}	0.93	2.81	3.728 (2)	169
C10—H10···O3	0.93	2.26	2.7009 (18)	108
C12—H12···O2	0.93	2.15	2.998 (2)	151
C14—H14···N1^{iii}	0.93	2.58	3.396 (2)	147
C17—H17*A*···O3^{iv}	0.96	2.78	3.615 (2)	147
C17—H17*C*···*Cg*^{iv}	0.96	2.82	3.606 (2)	139

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