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(*E*)-5-[1-Hydroxy-3-(3,4,5-trimethoxyphenyl)allylidene]-1,3-dimethylpyrimidine-2,4,6-trione: crystal structure and Hirshfeld surface analysis

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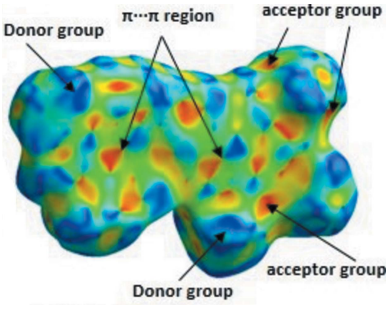
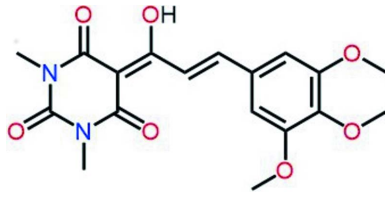
In the title compound, $C_{18}H_{20}N_2O_7$, the dihedral angle between the aromatic rings is 7.28 (7)° and the almost planar conformation of the molecule is supported by an intramolecular O—H···O hydrogen bond, which closes an *S*(6) ring. In the crystal, weak C—H···O hydrogen bonds and aromatic π–π stacking link the molecules into a three-dimensional network. A Hirshfeld surface analysis showed that the major contribution to the intermolecular interactions are van der Waals interactions (H···H contacts), accounting for 48.4% of the surface.

1. Chemical context

Bartituric acid derivatives are of interest due to their potential biological applications (Bojarski *et al.*, 1985; Patrick, 2009). These compounds have materials science appplications due to the properties generated by π-conjugation, such as push–pull chromophores (Klikar *et al.*, 2013; Seifert *et al.*, 2012). The chemical structures of these derivatives show five potential metal-binding sites, which makes them versatile ligands for the construction of coordination and supramolecular compounds (Mahmudov *et al.*, 2014), also important in organic synthesis, where they are largely used as substrates for Morita–Baylis–Hilmann and Diels–Alder reactions (Goswami & Das, 2009). Herein we report the crystal structure and Hisrshfeld surface analysis of (*E*)-5-[1-hydroxy-3-(3,4,5-trimethoxyphenyl)allylidene]-1,3-dimethylpyrimidine-2,4,6-trione (I), which presents potential applications in the study of the photophysical properties of different isomers for the development of supramolecular structures.





2. Structural commentary

The structure of (I), which crystallizes in the triclinic space group $P\overline{1}$, presents conjugation over the C1—C10—C11—C12—C13 bonds, leading to a an almost planar conformation (Fig. 1); the C10—C11—C12—C13 and C1—C10—C11—C12

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O5	0.82	1.74	2.4841 (15)	150
C11—H11···O7	0.93	2.16	2.8044 (18)	125
C8—H8B···O6^{i}	0.96	2.60	3.341 (3)	135
C9—H9C···O7^{ii}	0.96	2.42	3.3694 (19)	170

Symmetry codes: (i) $x, y, z+1$; (ii) $-x-1, -y+1, -z+2$.

torsion angles are −176.76 (1) and −179.27 (1)°, respectively. The dihedral angle between the aromatic rings is 7.28 (7)°. The C atoms of the *meta*-methoxy groups lie close to the plane of their attached ring [deviations for atoms C7 and C9 of 0.289 (2) and 0.131 (2)Å, respectively], whereas the *para*-methoxy C atom deviates significantly, by 0.959 (2) Å, which is reflected in the C3—C4—O2—C8 torsion angle of 106.41 (19)°. An intramolecular O—H···O hydrogen bond (Table 1) closes an S(6) ring and a C—H···O interaction is also observed. A *Mogul* geometry check found that all the bond lengths and angles are within typical ranges (Bruno *et al.*, 2004).

3. Supramolecular features

The packing of the title compound features inversion dimers linked by pairs of C9—H9C···O7^{ii} hydrogen bonds [C···O = 3.3694 (19) Å], which generate $R_2^2(24)$ loops. The dimers are linked along the [001] direction by the C8—H8B···O6^{i} hydrogen bond [C···O = 3.341 (3) Å]. In addition, weak and very weak π–π interactions (which alternate with respect to the [010] direction) between benzene and pyrimidine rings [centroid–centroid separations = 3.8779 (1) and 4.2283 (9) Å, respectively] occur (Fig. 2). Together, these intermolecular interactions lead to a three-dimensional network (Fig. 3).

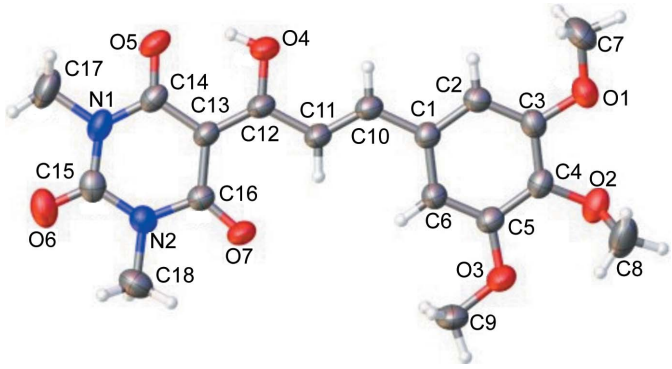


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

4. Hirshfeld surfaces analysis

The Hirshfeld surface analysis shows the potential intermolecular contacts. Convex blue regions represent hydrogen-donor groups and concave red regions represent hydrogen-acceptor groups (Hirshfeld, 1977; McKinnon *et al.*, 2004). In this case, the main donor groups are the methyl groups and the acceptor groups are the O atoms. The region of π–π inter-

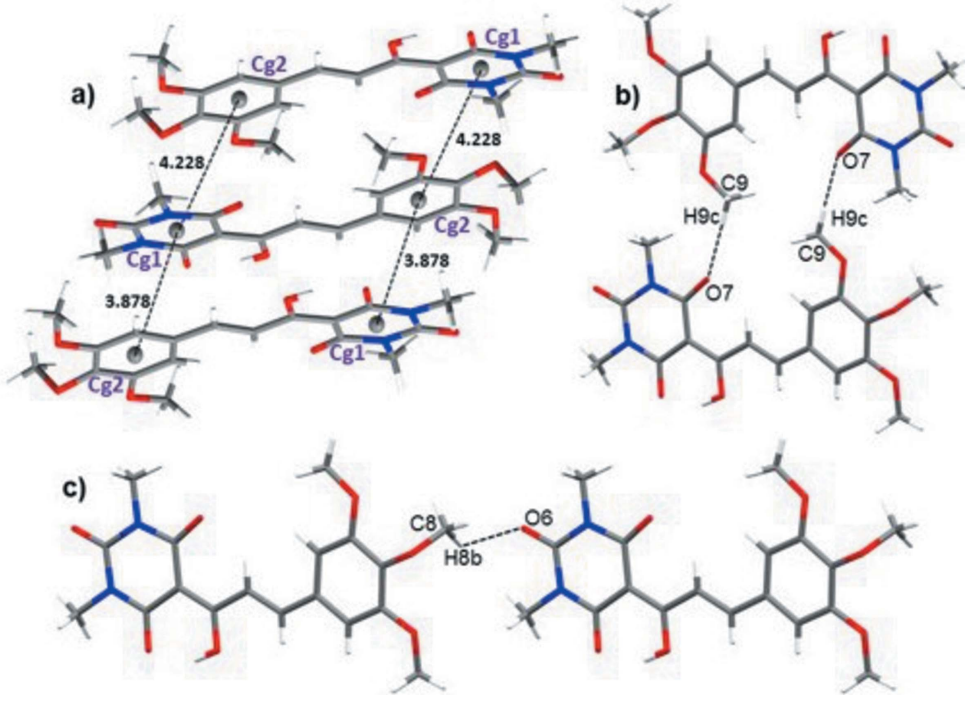


Figure 2
Details of the intermolecular interactions in the crystal of (I), showing (*a*) π–π stacking between rings 1 (N1/N2/C13–C16) and 2 (C1–C6) along the [010] direction, (*b*) an inversion dimer formed by the C9—H9C···O7^{ii} hydrogen bond and (*c*) by the C8—H8B···O6^{i} hydrogen bond. [Symmetry codes: (i) x, y, $1+z$; (ii) $-1-x$, $1-y$, $-z$.]

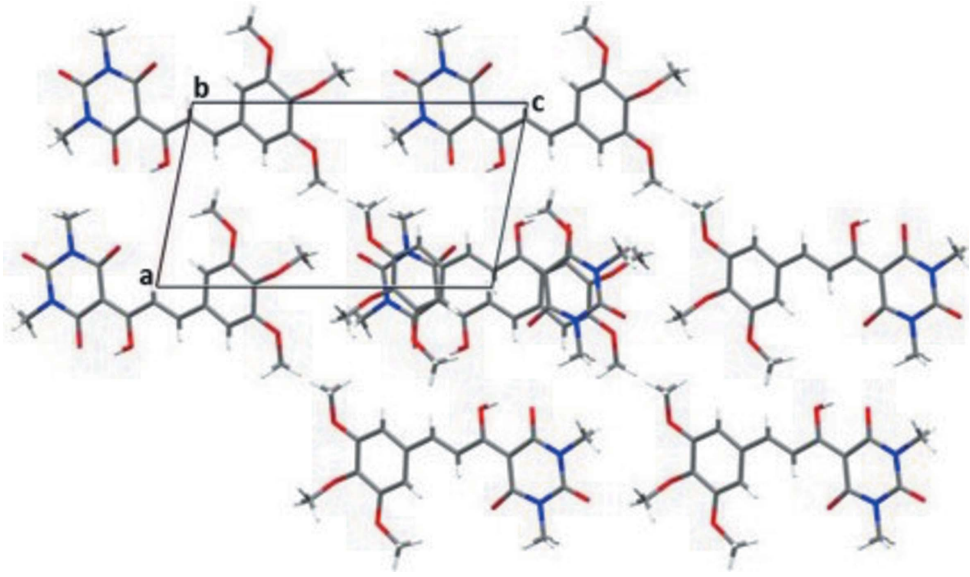


Figure 3
Crystal packing representation of (I), viewed along the [010] direction.

actions, observed as red and blue triangles over the aromatic rings, is also clear (Fig. 4). This surface confirms the importance of the interactions described previously.

The two-dimensional fingerprint plot quantifies the contribution of each kind of interaction to the surface formation (McKinnon *et al.*, 2007). For the title compound (Fig. 5), the major contribution is due to H···H corresponding to van der Waals interactions with 48.4% of the surface, followed by the O···H interaction, which contributes 26.5% (this contribution is observed as two sharp peaks in the plot); this behaviour is usual for strong hydrogen bonds (Spackman & McKinnon, 2002). Finally, π–π interactions represented by C···C interactions contribute 6.0% to the Hirshfeld surface.

5. Database survey

A general search in the Cambridge Structural Database (Groom *et al.*, 2016) for barbituric acid derivatives yielded 718 hits. Limiting the search for a barbituric acid substituted at position C5 with a phenylpropyl group yielded 14 hits; two of

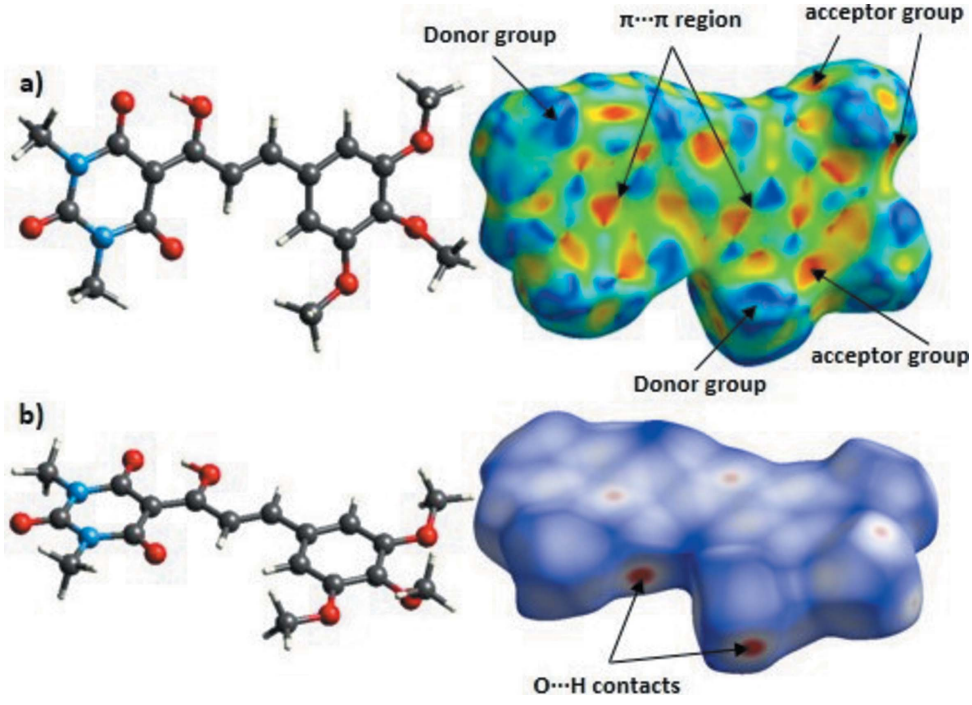


Figure 4
Hirshfeld surface of the title compound as (*a*) shape index and (*b*) d_{norm}.

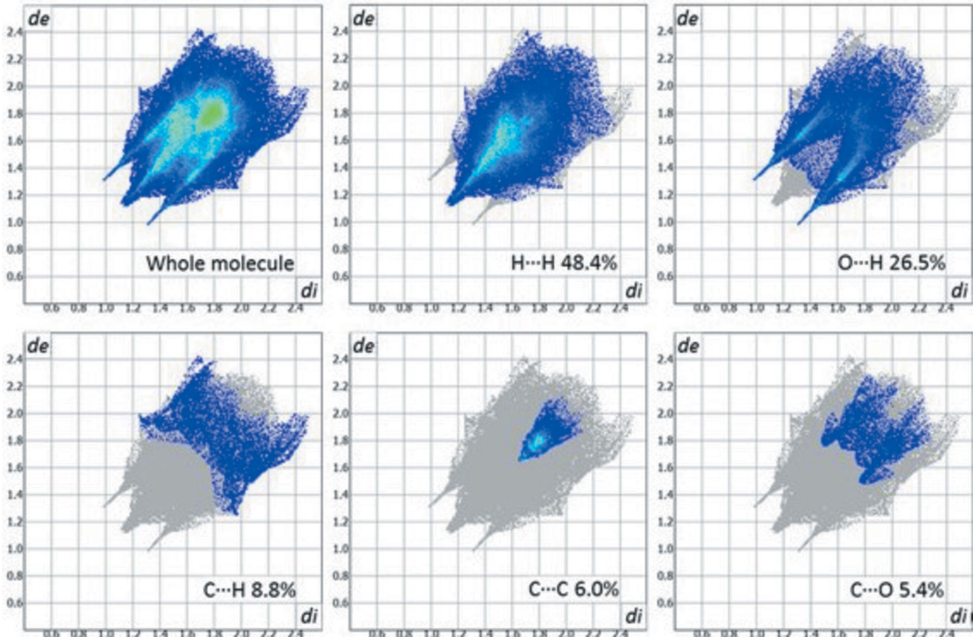


Figure 5
Bidimensional fingerprint plots for the whole molecule and H···H, O···H, C···H, C···C and C···O close contacts.

these results present double-bond conjugation, namely 1,3-dibutyl-5-{3-[4-(dimethylamino)phenyl]prop-2-en-1-ylidene}-pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (Klikar *et al.*, 2013) and 5-{3-[4-(dimethylamino)phenyl]prop-2-en-1-ylidene}pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (Seifert *et al.*, 2012).

6. Synthesis and crystallization

The title compound was prepared according to the literature procedure of Gorovoy *et al.* (2014). A mixture of 3,4,5-trimethoxybenzaldehyde and 5-acetyl-1,3-dimethylbarbituric acid was melted at 453 K and 2–3 drops of piperidine were added under constant stirring. After 5 min, the mixture solidified, providing a yellow powder, which was allowed to cool to room temperature. The solid residue was boiled in ethanol (20 ml) for a few minutes and the precipitate was filtered off by vacuum suction. The filtrate was left at room temperature, yielding yellow needles of the title compound after three weeks.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H-atom positions were calculated geometrically and refined using the riding model, with O—H = 0.82 Å, methyl C—H = 0.96 Å and aromatic C—H = 0.93 Å.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{20}N_2O_7$
M_r	376.36
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	7.9989 (3), 8.0659 (3), 14.6533 (5)
α, β, γ (°)	104.520 (1), 98.422 (1), 98.909 (1)
V (Å^3)	887.04 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	1.07 × 0.33 × 0.28
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.714, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26167, 3626, 3176
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.627
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.130, 1.04
No. of reflections	3626
No. of parameters	250
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *CrystalExplorer* (McKinnon *et al.*, 2004), *WinGX* (Farrugia, 2012) and *OLEX2* (Dolomanov *et al.*, 2009).

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(*E*)-5-[1-Hydroxy-3-(3,4,5-trimethoxyphenyl)allylidene]-1,3-dimethylpyrimidine-2,4,6-trione: crystal structure and Hirshfeld surface analysis

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Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *CrystalExplorer* (McKinnon *et al.*, 2004); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *OLEX2* (Dolomanov *et al.*, 2009).

(*E*)-5-[1-Hydroxy-3-(3,4,5-trimethoxyphenyl)allylidene]-1,3-dimethylpyrimidine-2,4,6-trione

Crystal data

$C_{18}H_{20}N_2O_7$
$M_r = 376.36$
Triclinic, $P\overline{1}$
$a = 7.9989$ (3) Å
$b = 8.0659$ (3) Å
$c = 14.6533$ (5) Å
$\alpha = 104.520$ (1)°
$\beta = 98.422$ (1)°
$\gamma = 98.909$ (1)°
$V = 887.04$ (6) Å^3
$Z = 2$
$F(000) = 396$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9895 reflections
$\theta = 2.6$–26.4°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Needle, light yellow
1.07 × 0.33 × 0.28 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: microfocus sealed X-ray tube
Detector resolution: 7.9 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
$T_{min} = 0.714$, $T_{max} = 0.745$
26167 measured reflections
3626 independent reflections
3176 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.5°$, $\theta_{min} = 1.5°$
$h = -10 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.130$
$S = 1.04$
3626 reflections
250 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.233P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.10573 (17)	0.71389 (17)	1.15734 (9)	0.0393 (3)
C2	0.21740 (18)	0.71690 (18)	1.24039 (10)	0.0430 (3)
H2	0.3327	0.7711	1.2512	0.052*
C3	0.15737 (19)	0.63927 (18)	1.30710 (10)	0.0435 (3)
C4	−0.01447 (19)	0.55537 (18)	1.29072 (10)	0.0426 (3)
C5	−0.12826 (18)	0.55865 (19)	1.20923 (10)	0.0446 (3)
C6	−0.06846 (18)	0.63736 (18)	1.14315 (10)	0.0433 (3)
H6	−0.1446	0.6392	1.0892	0.052*
C7	0.4228 (3)	0.7414 (3)	1.41741 (15)	0.0841 (7)
H7A	0.4901	0.7025	1.3701	0.126*
H7B	0.4774	0.7334	1.4786	0.126*
H7C	0.4144	0.8605	1.4220	0.126*
C8	−0.1648 (3)	0.5368 (3)	1.41581 (15)	0.0809 (6)
H8A	−0.2713	0.5483	1.3805	0.121*
H8B	−0.1021	0.6501	1.4544	0.121*
H8C	−0.1886	0.4634	1.4567	0.121*
C9	−0.4173 (2)	0.4882 (3)	1.12169 (14)	0.0699 (5)
H9A	−0.3869	0.4312	1.0624	0.105*
H9B	−0.4172	0.6085	1.1248	0.105*
H9C	−0.5302	0.4314	1.1248	0.105*
C10	0.17536 (17)	0.78939 (18)	1.08679 (10)	0.0422 (3)
H10	0.2915	0.8426	1.1020	0.051*
C11	0.08636 (18)	0.78836 (19)	1.00250 (10)	0.0443 (3)
H11	−0.0303	0.7369	0.9863	0.053*
C12	0.16329 (17)	0.86397 (18)	0.93467 (10)	0.0426 (3)
C13	0.07305 (17)	0.87319 (17)	0.84766 (10)	0.0392 (3)
C14	0.16886 (19)	0.94308 (18)	0.78507 (11)	0.0441 (3)
C15	−0.0952 (2)	0.88897 (19)	0.66628 (11)	0.0480 (3)
C16	−0.11265 (18)	0.81213 (18)	0.81775 (10)	0.0417 (3)
C17	0.1805 (3)	1.0066 (3)	0.63158 (14)	0.0735 (5)
H17A	0.2704	1.1046	0.6673	0.110*
H17B	0.1053	1.0413	0.5856	0.110*
H17C	0.2307	0.9140	0.5986	0.110*
C18	−0.3720 (2)	0.7703 (3)	0.69641 (14)	0.0674 (5)
H18A	−0.3975	0.6558	0.6518	0.101*
H18B	−0.4164	0.8507	0.6656	0.101*

H18C	−0.4248	0.7668	0.7509	0.101*
N1	0.08167 (17)	0.94549 (16)	0.69760 (9)	0.0479 (3)
N2	−0.18496 (15)	0.82765 (16)	0.72844 (9)	0.0463 (3)
O1	0.25706 (15)	0.63579 (17)	1.39048 (8)	0.0625 (3)
O2	−0.06606 (16)	0.46212 (14)	1.35161 (8)	0.0573 (3)
O3	−0.29489 (14)	0.47840 (17)	1.20048 (9)	0.0638 (3)
O4	0.33028 (13)	0.92287 (18)	0.96038 (8)	0.0611 (3)
H4	0.3661	0.9594	0.9180	0.092*
O5	0.32765 (14)	1.00179 (16)	0.80691 (9)	0.0603 (3)
O6	−0.16669 (18)	0.89585 (18)	0.58887 (8)	0.0693 (4)
O7	−0.20868 (13)	0.75253 (17)	0.86397 (8)	0.0608 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0395 (7)	0.0408 (7)	0.0361 (6)	0.0050 (5)	0.0078 (5)	0.0095 (5)
C2	0.0369 (7)	0.0482 (7)	0.0415 (7)	0.0046 (5)	0.0054 (5)	0.0117 (6)
C3	0.0455 (8)	0.0483 (7)	0.0378 (7)	0.0118 (6)	0.0055 (6)	0.0141 (6)
C4	0.0482 (8)	0.0422 (7)	0.0418 (7)	0.0091 (6)	0.0138 (6)	0.0164 (6)
C5	0.0393 (7)	0.0475 (7)	0.0455 (7)	0.0016 (6)	0.0092 (6)	0.0143 (6)
C6	0.0398 (7)	0.0524 (8)	0.0367 (7)	0.0038 (6)	0.0047 (5)	0.0151 (6)
C7	0.0596 (11)	0.1186 (17)	0.0669 (12)	−0.0029 (11)	−0.0179 (9)	0.0429 (12)
C8	0.0891 (14)	0.1140 (17)	0.0749 (13)	0.0458 (13)	0.0456 (11)	0.0575 (12)
C9	0.0363 (8)	0.0968 (14)	0.0761 (12)	−0.0053 (8)	0.0029 (8)	0.0393 (10)
C10	0.0353 (7)	0.0473 (7)	0.0413 (7)	0.0018 (5)	0.0082 (5)	0.0111 (6)
C11	0.0350 (7)	0.0537 (8)	0.0424 (7)	−0.0011 (6)	0.0078 (5)	0.0159 (6)
C12	0.0327 (6)	0.0493 (7)	0.0448 (7)	0.0012 (5)	0.0100 (5)	0.0142 (6)
C13	0.0354 (7)	0.0428 (7)	0.0409 (7)	0.0038 (5)	0.0119 (5)	0.0142 (5)
C14	0.0444 (8)	0.0436 (7)	0.0504 (8)	0.0080 (6)	0.0194 (6)	0.0186 (6)
C15	0.0592 (9)	0.0477 (8)	0.0417 (7)	0.0173 (6)	0.0125 (6)	0.0150 (6)
C16	0.0370 (7)	0.0455 (7)	0.0425 (7)	0.0053 (5)	0.0085 (5)	0.0138 (6)
C17	0.0860 (13)	0.0876 (13)	0.0673 (11)	0.0165 (11)	0.0381 (10)	0.0456 (10)
C18	0.0459 (9)	0.0906 (13)	0.0620 (10)	0.0097 (8)	−0.0030 (8)	0.0244 (9)
N1	0.0586 (8)	0.0482 (7)	0.0462 (7)	0.0127 (5)	0.0216 (6)	0.0224 (5)
N2	0.0426 (7)	0.0535 (7)	0.0431 (6)	0.0096 (5)	0.0064 (5)	0.0152 (5)
O1	0.0543 (7)	0.0855 (8)	0.0517 (6)	0.0084 (6)	0.0002 (5)	0.0352 (6)
O2	0.0710 (7)	0.0561 (6)	0.0581 (7)	0.0159 (5)	0.0247 (6)	0.0309 (5)
O3	0.0423 (6)	0.0871 (8)	0.0622 (7)	−0.0093 (5)	0.0059 (5)	0.0370 (6)
O4	0.0330 (5)	0.0924 (9)	0.0565 (7)	−0.0072 (5)	0.0047 (5)	0.0324 (6)
O5	0.0428 (6)	0.0774 (8)	0.0696 (7)	0.0017 (5)	0.0222 (5)	0.0362 (6)
O6	0.0839 (9)	0.0855 (9)	0.0461 (6)	0.0249 (7)	0.0099 (6)	0.0284 (6)
O7	0.0348 (5)	0.0905 (8)	0.0591 (7)	−0.0065 (5)	0.0078 (5)	0.0369 (6)

Geometric parameters (Å, º)

C1—C2	1.3926 (19)	C10—C11	1.330 (2)
C1—C6	1.3964 (19)	C10—H10	0.9300
C1—C10	1.4592 (19)	C11—C12	1.4509 (19)

C2—C3	1.389 (2)	C11—H11	0.9300
C2—H2	0.9300	C12—O4	1.3108 (17)
C3—O1	1.3668 (17)	C12—C13	1.395 (2)
C3—C4	1.392 (2)	C13—C14	1.4414 (18)
C4—O2	1.3713 (17)	C13—C16	1.4545 (19)
C4—C5	1.400 (2)	C14—O5	1.2484 (18)
C5—O3	1.3613 (17)	C14—N1	1.3728 (19)
C5—C6	1.3854 (19)	C15—O6	1.2115 (19)
C6—H6	0.9300	C15—N2	1.377 (2)
C7—O1	1.403 (2)	C15—N1	1.388 (2)
C7—H7A	0.9600	C16—O7	1.2171 (17)
C7—H7B	0.9600	C16—N2	1.3949 (18)
C7—H7C	0.9600	C17—N1	1.4651 (19)
C8—O2	1.397 (2)	C17—H17A	0.9600
C8—H8A	0.9600	C17—H17B	0.9600
C8—H8B	0.9600	C17—H17C	0.9600
C8—H8C	0.9600	C18—N2	1.464 (2)
C9—O3	1.427 (2)	C18—H18A	0.9600
C9—H9A	0.9600	C18—H18B	0.9600
C9—H9B	0.9600	C18—H18C	0.9600
C9—H9C	0.9600	O4—H4	0.8200

C2—C1—C6	119.47 (12)	C10—C11—H11	118.6
C2—C1—C10	118.69 (12)	C12—C11—H11	118.6
C6—C1—C10	121.83 (12)	O4—C12—C13	120.83 (12)
C3—C2—C1	120.32 (13)	O4—C12—C11	114.45 (12)
C3—C2—H2	119.8	C13—C12—C11	124.71 (12)
C1—C2—H2	119.8	C12—C13—C14	118.37 (12)
O1—C3—C2	124.48 (13)	C12—C13—C16	122.27 (12)
O1—C3—C4	115.30 (12)	C14—C13—C16	119.35 (13)
C2—C3—C4	120.22 (13)	O5—C14—N1	118.50 (12)
O2—C4—C3	119.14 (13)	O5—C14—C13	122.93 (14)
O2—C4—C5	121.34 (13)	N1—C14—C13	118.57 (13)
C3—C4—C5	119.39 (12)	O6—C15—N2	122.00 (15)
O3—C5—C6	124.28 (13)	O6—C15—N1	121.53 (15)
O3—C5—C4	115.49 (12)	N2—C15—N1	116.47 (13)
C6—C5—C4	120.22 (13)	O7—C16—N2	118.09 (13)
C5—C6—C1	120.23 (13)	O7—C16—C13	126.00 (13)
C5—C6—H6	119.9	N2—C16—C13	115.90 (12)
C1—C6—H6	119.9	N1—C17—H17A	109.5
O1—C7—H7A	109.5	N1—C17—H17B	109.5
O1—C7—H7B	109.5	H17A—C17—H17B	109.5
H7A—C7—H7B	109.5	N1—C17—H17C	109.5
O1—C7—H7C	109.5	H17A—C17—H17C	109.5
H7A—C7—H7C	109.5	H17B—C17—H17C	109.5
H7B—C7—H7C	109.5	N2—C18—H18A	109.5
O2—C8—H8A	109.5	N2—C18—H18B	109.5
O2—C8—H8B	109.5	H18A—C18—H18B	109.5

H8A—C8—H8B	109.5	N2—C18—H18C	109.5
O2—C8—H8C	109.5	H18A—C18—H18C	109.5
H8A—C8—H8C	109.5	H18B—C18—H18C	109.5
H8B—C8—H8C	109.5	C14—N1—C15	123.97 (12)
O3—C9—H9A	109.5	C14—N1—C17	118.57 (14)
O3—C9—H9B	109.5	C15—N1—C17	117.44 (14)
H9A—C9—H9B	109.5	C15—N2—C16	125.68 (13)
O3—C9—H9C	109.5	C15—N2—C18	116.73 (13)
H9A—C9—H9C	109.5	C16—N2—C18	117.58 (13)
H9B—C9—H9C	109.5	C3—O1—C7	117.14 (13)
C11—C10—C1	125.35 (13)	C4—O2—C8	116.71 (13)
C11—C10—H10	117.3	C5—O3—C9	117.32 (12)
C1—C10—H10	117.3	C12—O4—H4	109.5
C10—C11—C12	122.86 (13)		

C6—C1—C2—C3	2.1 (2)	C16—C13—C14—N1	−1.7 (2)
C10—C1—C2—C3	−177.07 (13)	C12—C13—C16—O7	2.1 (2)
C1—C2—C3—O1	−179.95 (13)	C14—C13—C16—O7	−179.05 (15)
C1—C2—C3—C4	1.0 (2)	C12—C13—C16—N2	−179.24 (12)
O1—C3—C4—O2	−6.8 (2)	C14—C13—C16—N2	−0.35 (19)
C2—C3—C4—O2	172.34 (13)	O5—C14—N1—C15	−178.00 (13)
O1—C3—C4—C5	177.24 (13)	C13—C14—N1—C15	1.9 (2)
C2—C3—C4—C5	−3.6 (2)	O5—C14—N1—C17	3.8 (2)
O2—C4—C5—O3	6.2 (2)	C13—C14—N1—C17	−176.34 (14)
C3—C4—C5—O3	−177.92 (13)	O6—C15—N1—C14	179.01 (14)
O2—C4—C5—C6	−172.77 (13)	N2—C15—N1—C14	0.1 (2)
C3—C4—C5—C6	3.1 (2)	O6—C15—N1—C17	−2.7 (2)
O3—C5—C6—C1	−178.84 (14)	N2—C15—N1—C17	178.30 (14)
C4—C5—C6—C1	0.0 (2)	O6—C15—N2—C16	178.65 (14)
C2—C1—C6—C5	−2.7 (2)	N1—C15—N2—C16	−2.4 (2)
C10—C1—C6—C5	176.53 (13)	O6—C15—N2—C18	0.1 (2)
C2—C1—C10—C11	176.58 (14)	N1—C15—N2—C18	179.10 (14)
C6—C1—C10—C11	−2.6 (2)	O7—C16—N2—C15	−178.70 (14)
C1—C10—C11—C12	−179.27 (13)	C13—C16—N2—C15	2.5 (2)
C10—C11—C12—O4	4.4 (2)	O7—C16—N2—C18	−0.2 (2)
C10—C11—C12—C13	−176.76 (14)	C13—C16—N2—C18	−179.01 (14)
O4—C12—C13—C14	2.3 (2)	C2—C3—O1—C7	10.7 (2)
C11—C12—C13—C14	−176.48 (13)	C4—C3—O1—C7	−170.18 (17)
O4—C12—C13—C16	−178.81 (13)	C3—C4—O2—C8	106.41 (19)
C11—C12—C13—C16	2.4 (2)	C5—C4—O2—C8	−77.7 (2)
C12—C13—C14—O5	−2.9 (2)	C6—C5—O3—C9	−4.9 (2)
C16—C13—C14—O5	178.20 (13)	C4—C5—O3—C9	176.16 (15)
C12—C13—C14—N1	177.25 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O5	0.82	1.74	2.4841 (15)	150

C11—H11···O7	0.93	2.16	2.8044 (18)	125
C8—H8*B*···O6^{i}	0.96	2.60	3.341 (3)	135
C9—H9*C*···O7^{ii}	0.96	2.42	3.3694 (19)	170

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