Crystal structure of (*E*)-3-(2-hydroxy-4-methyl-phenyl)-1-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

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The title chalcone derivative, $C_{19}H_{20}O_5$, adopts a *trans* configuration with respect to the olefinic C=C double bond. The 2-hydroxy-4-methylphenyl ring is coplanar with the attached enone bridge [torsion angle = −179.96 (14)°], where this plane is nearly perpendicular to the 2,4,6-trimethoxyphenyl ring [dihedral angle = 75.81 (8)°]. In the crystal, molecules are linked into chains propagating along [010] by an O—H···O hydrogen bond. These chains are further connected into centrosymmetric dimer chains *via* weak C—H···O interactions. The conformations of related chalcone derivatives are surveyed and all of these structures adopt a skeleton with two almost orthogonal aromatic rings.

1. Chemical context

Chalcones (1,3-diarylprop-2-en-1-ones) are precursors of flavonoids and isoflavonoids in the plant kingdom (Ni *et al.*, 2004; Sahu *et al.*, 2012). Structurally, they consist of two aryl groups linked by an α, β-unsaturated ketone system (Ibrahim *et al.*, 2012; Kumar *et al.*, 2013), whereby the aryl groups can carry a variety of substituents such as hydroxyl, methoxy and alkenyl groups, which are by far the most commonly encountered ones in nature. With their structural simplicity and the associated ease of synthesis, chalcone compounds have attracted a considerable amount of attention because of their important pharmacological properties such as anti-oxidative (Aoki *et al.*, 2008), anti-inflammatory (Israf *et al.*, 2007), anti-gout (Jang *et al.*, 2014), anti-histaminic (Yamamoto *et al.*, 2004), anti-obesity (Birari *et al.*, 2011), anti-protozoal (Chen *et al.*, 1993), hypnotic (Cho *et al.*, 2011) and anti-spasmodic (Sato *et al.*, 2007) effects. In a continuation of our ongoing research on the properties of various chalcone derivatives (Sim *et al.*, 2017, Kwong *et al.*, 2018), we report herein the synthesis and crystal structure determination of the title compound, $C_{19}H_{20}O_5$, (I).

2. Structural commentary

The title chalcone derivative (I), crystallizes in the centrosymmetric triclinic space group $P\bar{1}$ and its asymmetric unit
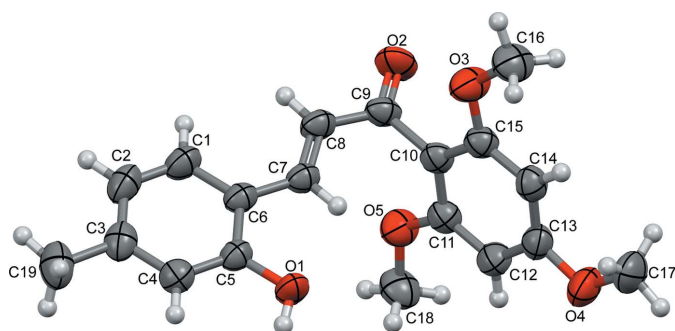
Figure 1
The title molecule with the atom-labelling scheme and displacement ellipsoids drawn at the 50% ellipsoid probability level.

consists of a single unique molecule (Fig. 1). This molecule is constructed of two substituted aromatic rings (2-hydroxy-4-methylphenyl and 2,4,6-trimethoxyphenyl) and an enone (–CH=CH–(C=O)–) connecting bridge. In the enone bridge, the C6—C7, C8—C9 and C9—C10 bond lengths of 1.446 (2), 1.441 (2) and 1.504 (2) Å, respectively, confirm their single-bond character, whereas the C7=C8 and C9=O2 bond lengths of 1.340 (2) and 1.2255 (17) Å, respectively, confirm the presence of a double bond. In addition, the C6—C7—C8 and C8—C9—C10 bond angles of 128.71 (13) and 119.47 (11)°, respectively, are consistent with the sp^2 hybridization of atoms C7, C8 and C9 (Kerr *et al.*, 2001; Loghmani-Khouzani *et al.*, 2009; Grealis *et al.*, 2013). As the olefinic double bond C7=C8 adopts a *trans* configuration [C6—C7—C8—C9 torsion angle = −179.96 (14)°], the structural conformation of (I) can be defined by three torsion angles. The torsion angles between the 2-hydroxy-4-methylphenyl ring and the olefinic double bond (C5—C6—C7—C8, τ_1), between the olefinic double bond and the carbonyl group (C7—C8—C9—C10, τ_2) and between the carbonyl group and the 2,4,6-trimethoxyphenyl ring (C8—C9—C10—C11, τ_3) are shown in Fig. 2. The torsion angles τ_1 and τ_2 are approximately ±180° or 0° [τ_1 = −179.15 (14)° and τ_2 = −0.8 (2)°], indicating that the 2-hydroxy-4-methylphenyl ring and the enone bridge are coplanar. In contrast, the carbonyl group is nearly perpendicular to the attached 2,4,6-trimethoxyphenyl ring, as τ_3 is 76.87 (19)°. In general, the molecule of (I) can be
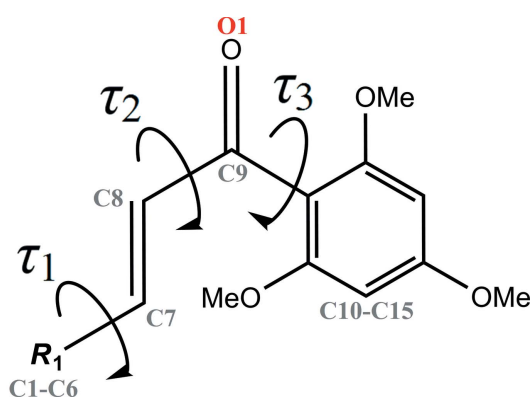
considered as two individual planes, the first comprising the 2-hydroxy-4-methylphenyl ring and the enone bridge [maximum deviation of 0.0021 (2) Å for atom C19], and the second the 2,4,6-trimethoxyphenyl ring [maximum deviation of 0.0059 (2) Å for atom C18]. These two mean planes form a dihedral angle of 75.84 (4)°, hence the molecule of (I) possesses a skeleton with two almost orthogonal aromatic rings.

3. Supramolecular features

In the crystal, the molecules are linked into chains parallel to the *b* axis *via* classical O1—H1*B*···O2^i hydrogen bonds (Fig. 3*a*). These chains are further connected into inversion-related dimeric chains by weak C17—H17*A*···O1^{ii} intermolecular interactions (Fig. 3*b*, Table 1).

4. Database survey

A search of the Cambridge Structural Database (CSD version 5.40, last update May 2019; Groom *et al.*, 2016) using (*E*)-3-phenyl-1-(2,4,6-trimethoxyphenyl)prop-2-en-1-one as the reference moiety resulted in three chalcone structures containing 2,4,6-trimethoxyphenyl with different substituents.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*B*···O2^i	0.82	1.88	2.6653 (15)	161
C17—H17*A*···O1^{ii}	0.96	2.70	3.520 (2)	144

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

Figure 2
General chemical diagram showing torsion angles, τ_1, τ_2 and τ_3.

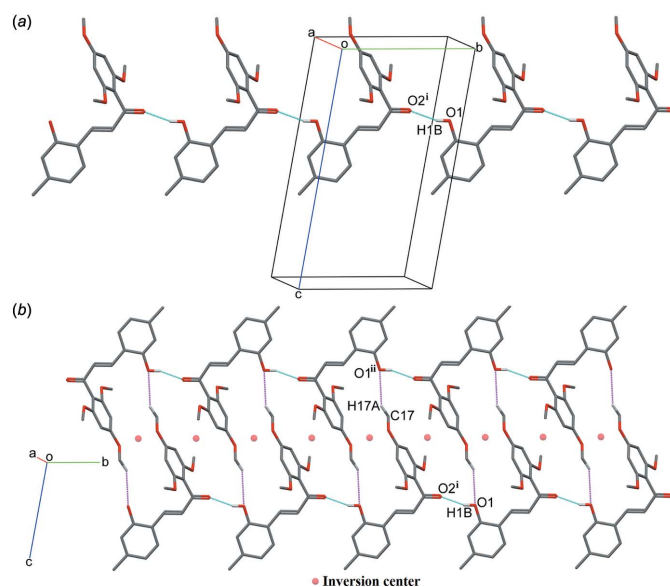**Figure 3**
Views of (*a*) a chain of molecules linked by O—H···O hydrogen bonds (shown as cyan dotted lines) and (*b*) a dimeric chain formed by weak C—H···O interactions (shown as magenta dotted lines). Symmetry code: (i) $x, -1 + y, z$; (ii) $1 - x, -y, -z$. Hydrogen atoms not involved in these interactions are omitted for clarity.

Table 2
Selected torsion and dihedral angles (°).

The dihedral angle is that between the mean planes of the aromatic rings.

Compound	R_1	τ_1 (C5—C6—C7—C8)	τ_2 (C7—C8—C9—C10)	τ_3 (C8—C9—C10—C11)	Dihedral angle
(I)	2-hydroxy-4-methylphenyl	−179.2 (1)	−0.8 (2)	76.9 (2)	75.8 (1)
BAGXEN	2,4,6-trimethoxyphenyl	174.1	−4.8	88.6	80.4
BUFMOF	6-nitrobenzo[d][1,3]dioxol-5-yl	153.7	6.8	67.6	61.6
GESRAZ	4-methoxyphenyl	176.0	7.6	−82.2	79.0

Note: values for the minor occupancy component of GESRAZ are not given.

They include (E)-3-(R_1)-1-(2,4,6-trimethoxyphenyl)prop-2-en-1-one, where R_1 = 2,4,6-trimethoxyphenyl (BAGXEN; Kerr *et al.*, 2001), 6-nitrobenzo[d][1,3]dioxol-5-yl (BUFMOF; Loghmani-Khouzani *et al.*, 2009) and 4-methoxyphenyl (GESRAZ; Grealis *et al.*, 2013). As in (I), the molecules of all these structures adopt a *trans* configuration with respect to C=C double bond (C6—C7—C8—C9 torsion angles = 175.5–179.1°). Although, τ_1 for all of the structures indicates an *anti-periplanar* conformation (Table 2), in BUFMOF it deviates slightly from planarity (τ_1 = 152.7°) whereas τ_1 for the other molecules is approximately 180° (τ_1 = 174.1–176.0°, Table 2). Regarding the enone bridge, the torsion angle τ_2 indicates that all of the structures are relatively planar (τ_2 = −4.8–7.6°). The torsion angles τ_3 always almost indicate a perpendicular arrangement (τ_3 = 67.6–88.6°). This might arise from the steric repulsion between the carbonyl group and the attached 2,4,6-trimethoxyphenyl ring. This results in an overall L-shape for all of the structures, with the dihedral angle between the mean planes of the two aromatic rings being 61.6–80.4°.

5. Synthesis and crystallization

A reaction scheme for the synthesis of the title compound is given in Fig. 4. A solution of trimethoxyacetophenone (2 mmol) in 20 mL MeOH, LiOH (2.4 mmol) and 2-hydroxy-4-methylbenzaldehyde (1.6 mmol) was stirred at 368 K and the reaction progress was monitored by TLC. The reaction was quenched with diluted hydrochloric acid to pH = 6 and extracted with ethyl acetate. The organic layer was washed with aqueous NaHCO₃, water, and brine, successively. The organic layer was dried over anhydrous Na₂SO₄ and then concentrated to provide the product as a brown powder. The residue was purified by column chromatography with petroleum ether/ethyl acetate (10:1) as an eluent to afford the target compound (Yan *et al.*, 2016). Slow evaporation from an acetonitrile–water mixture provided X-ray quality crystals for the target chalcone compound.
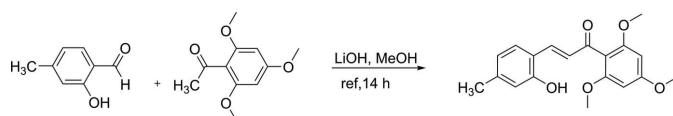

Figure 4
Reaction scheme for the synthesis of the title chalcone.

(E)-3-(2-hydroxy-4-methylphenyl)-1-(2,4,6-trimethoxy-phenyl)prop-2-en-1-one (I)

Brown powder, yield 84.1%. m.p. 503–506 K. IR (cm⁻¹): 3283 (O—H), 2929 and 2842 (Csp^3—H), 1603 (C=O), 1586 and 1457 (C=C). ¹H NMR (500 MHz, DMSO-d_6) δ, ppm: 10.02 (s, 1H), 7.45 (d, J = 16.2 Hz, 1H), 7.44 (d, J = 7.0 Hz, 1H), 6.86 (d, J = 16.2 Hz, 1H), 6.65 (d, J = 8.0 Hz, 2H), 6.30 (s, 2H), 3.83 (s, 3H), 3.70 (s, 6H), 2.23 (s, 3H). ¹³C-NMR (125 MHz, DMSO-d_6) δ, ppm: 194.3, 162.2, 158.4, 157.1, 142.5, 140.2, 128.8, 127.9, 121.1, 118.9, 117.0, 111.9, 91.6, 56.2, 55.9, 21.6. CHN Elemental analysis: Calculated for C₁₉H₂₀O₅: C, 69.50; H, 6.14; N. Found: C, 67.81; H, 5.72; N, 0.00.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned

Table 3
Experimental details.

Crystal data	
Chemical formula	C₁₉H₂₀O₅
M_r	328.35
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	296
a, b, c (Å)	6.8072 (3), 8.5792 (4), 15.8010 (7)
α, β, γ (°)	100.365 (1), 99.433 (1), 104.984 (1)
V (Å³)	855.09 (7)
Z	2
Radiation type	Mo $K\alpha$
μ (mm⁻¹)	0.09
Crystal size (mm)	0.57 × 0.25 × 0.21
Data collection	
Diffractometer	Bruker APEXII DUO CCD area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.908, 0.950
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33448, 5018, 3199
R_{int}	0.030
$(\sin \theta/\lambda)_{max}$ (Å⁻¹)	0.706
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.171, 1.05
No. of reflections	5018
No. of parameters	217
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.27, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2009).

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supporting information

Crystal structure of (*E*)-3-(2-hydroxy-4-methylphenyl)-1-(2,4,6-trimethoxy-phenyl)prop-2-en-1-one

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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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *SHELXL2013* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(*E*)-3-(2-Hydroxy-4-methylphenyl)-1-(2,4,6-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{19}H_{20}O_5$	$Z = 2$
$M_r = 328.35$	$F(000) = 348$
Triclinic, $P\overline{1}$	$D_x = 1.275$ Mg m^{-3}
$a = 6.8072$ (3) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.5792$ (4) Å	Cell parameters from 6606 reflections
$c = 15.8010$ (7) Å	$\theta = 2.5–25.7°$
$\alpha = 100.365$ (1)°	$\mu = 0.09$ mm^{-1}
$\beta = 99.433$ (1)°	$T = 296$ K
$\gamma = 104.984$ (1)°	Block, brown
$V = 855.09$ (7) Å^3	$0.57 \times 0.25 \times 0.21$ mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer	33448 measured reflections
Radiation source: fine-focus sealed tube	5018 independent reflections
Graphite monochromator	3199 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.030$
Absorption correction: multi-scan (SADABS; Bruker, 2012)	$\theta_{max} = 30.1°$, $\theta_{min} = 1.3°$
	$h = -9 \rightarrow 9$
	$k = -12 \rightarrow 12$
$T_{min} = 0.908$, $T_{max} = 0.950$	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.171$	$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.1331P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
5018 reflections	$(\Delta/\sigma)_{max} < 0.001$
217 parameters	$\Delta\rho_{max} = 0.27$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ 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}
O1	0.64130 (18)	0.04792 (12)	0.33273 (7)	0.0582 (3)
H1B	0.622234	−0.051206	0.330607	0.087*
O2	0.5594 (2)	0.71937 (13)	0.28620 (9)	0.0750 (4)
O3	0.82818 (19)	0.58546 (15)	0.17162 (8)	0.0718 (4)
O4	0.3336 (2)	0.11733 (16)	−0.04471 (7)	0.0741 (4)
O5	0.18978 (18)	0.33623 (15)	0.22860 (8)	0.0653 (3)
C1	0.7621 (3)	0.3898 (2)	0.52362 (11)	0.0587 (4)
H1A	0.765072	0.500561	0.536913	0.070*
C2	0.8168 (3)	0.3175 (2)	0.59089 (11)	0.0636 (4)
H2A	0.856694	0.379893	0.648810	0.076*
C3	0.8134 (2)	0.1518 (2)	0.57349 (10)	0.0576 (4)
C4	0.7548 (2)	0.0621 (2)	0.48690 (10)	0.0536 (4)
H4A	0.752792	−0.048531	0.474238	0.064*
C5	0.6988 (2)	0.13422 (17)	0.41849 (9)	0.0462 (3)
C6	0.7017 (2)	0.30075 (17)	0.43524 (9)	0.0461 (3)
C7	0.6399 (2)	0.37021 (17)	0.36199 (9)	0.0462 (3)
H7A	0.600214	0.297697	0.306638	0.055*
C8	0.6322 (2)	0.52507 (17)	0.36314 (10)	0.0530 (4)
H8A	0.670606	0.601882	0.417082	0.064*
C9	0.5668 (2)	0.57758 (17)	0.28428 (10)	0.0506 (3)
C10	0.5066 (2)	0.45590 (17)	0.19626 (10)	0.0474 (3)
C11	0.3145 (2)	0.33383 (18)	0.16927 (10)	0.0496 (3)
C12	0.2590 (2)	0.2222 (2)	0.08827 (10)	0.0553 (4)
H12A	0.130137	0.141133	0.070877	0.066*
C13	0.3996 (3)	0.23348 (19)	0.03331 (10)	0.0539 (4)
C14	0.5911 (2)	0.35440 (19)	0.05781 (10)	0.0528 (4)
H14A	0.683280	0.361673	0.020255	0.063*
C15	0.6426 (2)	0.46422 (18)	0.13936 (10)	0.0502 (3)
C16	0.9764 (3)	0.6078 (3)	0.11824 (14)	0.0747 (5)
H16A	1.098130	0.697163	0.149806	0.112*
H16B	1.014327	0.507669	0.103655	0.112*
H16C	0.917128	0.633866	0.065061	0.112*
C17	0.4721 (4)	0.1217 (3)	−0.10396 (12)	0.0798 (6)
H17A	0.408006	0.034279	−0.156077	0.120*
H17B	0.501338	0.226820	−0.119882	0.120*
H17C	0.599756	0.106887	−0.075655	0.120*
C18	−0.0021 (3)	0.2085 (3)	0.21029 (14)	0.0741 (5)
H18A	−0.072792	0.225952	0.257352	0.111*
H18B	−0.087708	0.210054	0.155938	0.111*

H18C	0.024368	0.102923	0.205183	0.111*
C19	0.8715 (3)	0.0699 (3)	0.64692 (12)	0.0785 (6)
H19A	0.908022	0.148913	0.702583	0.118*
H19B	0.755129	−0.021899	0.646508	0.118*
H19C	0.988302	0.030616	0.638320	0.118*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0834 (8)	0.0397 (5)	0.0461 (6)	0.0191 (5)	0.0077 (5)	0.0007 (4)
O2	0.1156 (11)	0.0394 (6)	0.0798 (8)	0.0294 (6)	0.0388 (8)	0.0135 (5)
O3	0.0660 (7)	0.0665 (7)	0.0692 (8)	−0.0007 (6)	0.0305 (6)	−0.0029 (6)
O4	0.0803 (9)	0.0796 (8)	0.0461 (6)	0.0084 (7)	0.0123 (6)	−0.0022 (6)
O5	0.0577 (7)	0.0702 (7)	0.0659 (7)	0.0118 (5)	0.0271 (6)	0.0090 (6)
C1	0.0631 (9)	0.0534 (8)	0.0508 (8)	0.0140 (7)	0.0116 (7)	−0.0047 (7)
C2	0.0594 (9)	0.0766 (11)	0.0441 (8)	0.0141 (8)	0.0094 (7)	−0.0018 (7)
C3	0.0434 (8)	0.0790 (11)	0.0486 (8)	0.0153 (7)	0.0102 (6)	0.0146 (7)
C4	0.0527 (8)	0.0538 (8)	0.0543 (8)	0.0169 (7)	0.0119 (7)	0.0113 (7)
C5	0.0454 (7)	0.0463 (7)	0.0431 (7)	0.0118 (6)	0.0099 (6)	0.0030 (6)
C6	0.0432 (7)	0.0440 (7)	0.0464 (7)	0.0097 (5)	0.0117 (6)	0.0018 (6)
C7	0.0476 (7)	0.0406 (7)	0.0466 (7)	0.0112 (5)	0.0130 (6)	0.0009 (5)
C8	0.0607 (9)	0.0413 (7)	0.0523 (8)	0.0129 (6)	0.0163 (7)	−0.0009 (6)
C9	0.0586 (8)	0.0357 (6)	0.0600 (9)	0.0137 (6)	0.0246 (7)	0.0074 (6)
C10	0.0557 (8)	0.0403 (7)	0.0507 (8)	0.0185 (6)	0.0173 (6)	0.0108 (6)
C11	0.0525 (8)	0.0491 (8)	0.0530 (8)	0.0198 (6)	0.0169 (6)	0.0152 (6)
C12	0.0529 (8)	0.0563 (9)	0.0522 (8)	0.0117 (7)	0.0082 (7)	0.0113 (7)
C13	0.0638 (9)	0.0550 (8)	0.0426 (7)	0.0206 (7)	0.0073 (7)	0.0103 (6)
C14	0.0597 (9)	0.0568 (8)	0.0475 (8)	0.0214 (7)	0.0192 (7)	0.0136 (7)
C15	0.0539 (8)	0.0446 (7)	0.0543 (8)	0.0156 (6)	0.0169 (7)	0.0113 (6)
C16	0.0663 (11)	0.0762 (12)	0.0799 (13)	0.0099 (9)	0.0345 (10)	0.0137 (10)
C17	0.1025 (15)	0.0791 (12)	0.0497 (10)	0.0168 (11)	0.0256 (10)	0.0018 (8)
C18	0.0613 (10)	0.0826 (13)	0.0820 (13)	0.0132 (9)	0.0271 (9)	0.0297 (10)
C19	0.0684 (11)	0.1119 (16)	0.0570 (11)	0.0267 (11)	0.0087 (9)	0.0291 (10)

Geometric parameters (Å, º)

O1—C5	1.3613 (16)	C8—H8A	0.9300
O1—H1B	0.8200	C9—C10	1.504 (2)
O2—C9	1.2255 (17)	C10—C15	1.390 (2)
O3—C15	1.3626 (19)	C10—C11	1.392 (2)
O3—C16	1.4151 (19)	C11—C12	1.383 (2)
O4—C13	1.3636 (18)	C12—C13	1.391 (2)
O4—C17	1.432 (2)	C12—H12A	0.9300
O5—C11	1.3651 (18)	C13—C14	1.385 (2)
O5—C18	1.419 (2)	C14—C15	1.384 (2)
C1—C2	1.371 (2)	C14—H14A	0.9300
C1—C6	1.403 (2)	C16—H16A	0.9600
C1—H1A	0.9300	C16—H16B	0.9600

C2—C3	1.392 (3)	C16—H16C	0.9600
C2—H2A	0.9300	C17—H17A	0.9600
C3—C4	1.381 (2)	C17—H17B	0.9600
C3—C19	1.511 (2)	C17—H17C	0.9600
C4—C5	1.387 (2)	C18—H18A	0.9600
C4—H4A	0.9300	C18—H18B	0.9600
C5—C6	1.3998 (19)	C18—H18C	0.9600
C6—C7	1.446 (2)	C19—H19A	0.9600
C7—C8	1.340 (2)	C19—H19B	0.9600
C7—H7A	0.9300	C19—H19C	0.9600
C8—C9	1.441 (2)		
C5—O1—H1B	109.5	C11—C12—C13	118.68 (15)
C15—O3—C16	119.21 (13)	C11—C12—H12A	120.7
C13—O4—C17	117.54 (14)	C13—C12—H12A	120.7
C11—O5—C18	118.63 (14)	O4—C13—C14	123.74 (14)
C2—C1—C6	121.65 (15)	O4—C13—C12	114.86 (14)
C2—C1—H1A	119.2	C14—C13—C12	121.39 (14)
C6—C1—H1A	119.2	C15—C14—C13	118.62 (14)
C1—C2—C3	120.75 (15)	C15—C14—H14A	120.7
C1—C2—H2A	119.6	C13—C14—H14A	120.7
C3—C2—H2A	119.6	O3—C15—C14	123.97 (13)
C4—C3—C2	118.51 (15)	O3—C15—C10	114.45 (13)
C4—C3—C19	120.07 (17)	C14—C15—C10	121.57 (14)
C2—C3—C19	121.41 (16)	O3—C16—H16A	109.5
C3—C4—C5	121.08 (15)	O3—C16—H16B	109.5
C3—C4—H4A	119.5	H16A—C16—H16B	109.5
C5—C4—H4A	119.5	O3—C16—H16C	109.5
O1—C5—C4	121.92 (13)	H16A—C16—H16C	109.5
O1—C5—C6	117.19 (12)	H16B—C16—H16C	109.5
C4—C5—C6	120.89 (13)	O4—C17—H17A	109.5
C5—C6—C1	117.12 (14)	O4—C17—H17B	109.5
C5—C6—C7	118.98 (12)	H17A—C17—H17B	109.5
C1—C6—C7	123.89 (13)	O4—C17—H17C	109.5
C8—C7—C6	128.71 (13)	H17A—C17—H17C	109.5
C8—C7—H7A	115.6	H17B—C17—H17C	109.5
C6—C7—H7A	115.6	O5—C18—H18A	109.5
C7—C8—C9	122.77 (13)	O5—C18—H18B	109.5
C7—C8—H8A	118.6	H18A—C18—H18B	109.5
C9—C8—H8A	118.6	O5—C18—H18C	109.5
O2—C9—C8	122.17 (14)	H18A—C18—H18C	109.5
O2—C9—C10	118.36 (14)	H18B—C18—H18C	109.5
C8—C9—C10	119.47 (12)	C3—C19—H19A	109.5
C15—C10—C11	118.35 (13)	C3—C19—H19B	109.5
C15—C10—C9	120.27 (13)	H19A—C19—H19B	109.5
C11—C10—C9	121.37 (13)	C3—C19—H19C	109.5
O5—C11—C12	124.18 (14)	H19A—C19—H19C	109.5
O5—C11—C10	114.43 (13)	H19B—C19—H19C	109.5

C12—C11—C10	121.39 (14)		
C6—C1—C2—C3	−0.3 (3)	C18—O5—C11—C12	5.6 (2)
C1—C2—C3—C4	0.4 (2)	C18—O5—C11—C10	−174.96 (14)
C1—C2—C3—C19	−179.49 (16)	C15—C10—C11—O5	−179.09 (12)
C2—C3—C4—C5	−0.5 (2)	C9—C10—C11—O5	0.4 (2)
C19—C3—C4—C5	179.37 (14)	C15—C10—C11—C12	0.4 (2)
C3—C4—C5—O1	179.78 (14)	C9—C10—C11—C12	179.85 (13)
C3—C4—C5—C6	0.5 (2)	O5—C11—C12—C13	179.48 (13)
O1—C5—C6—C1	−179.66 (13)	C10—C11—C12—C13	0.1 (2)
C4—C5—C6—C1	−0.3 (2)	C17—O4—C13—C14	0.2 (2)
O1—C5—C6—C7	1.36 (19)	C17—O4—C13—C12	−179.57 (15)
C4—C5—C6—C7	−179.32 (13)	C11—C12—C13—O4	179.14 (13)
C2—C1—C6—C5	0.2 (2)	C11—C12—C13—C14	−0.6 (2)
C2—C1—C6—C7	179.15 (14)	O4—C13—C14—C15	−178.99 (14)
C5—C6—C7—C8	−179.68 (14)	C12—C13—C14—C15	0.8 (2)
C1—C6—C7—C8	1.4 (2)	C16—O3—C15—C14	3.0 (2)
C6—C7—C8—C9	−179.96 (14)	C16—O3—C15—C10	−178.38 (15)
C7—C8—C9—O2	179.67 (15)	C13—C14—C15—O3	178.26 (14)
C7—C8—C9—C10	−0.8 (2)	C13—C14—C15—C10	−0.3 (2)
O2—C9—C10—C15	75.90 (19)	C11—C10—C15—O3	−178.96 (13)
C8—C9—C10—C15	−103.68 (16)	C9—C10—C15—O3	1.6 (2)
O2—C9—C10—C11	−103.56 (17)	C11—C10—C15—C14	−0.3 (2)
C8—C9—C10—C11	76.87 (19)	C9—C10—C15—C14	−179.73 (13)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1B⋯O2^i	0.82	1.88	2.6653 (15)	161
C17—H17A⋯O1^{ii}	0.96	2.70	3.520 (2)	144

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