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

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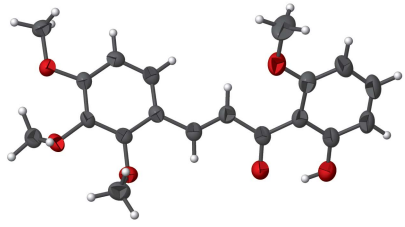
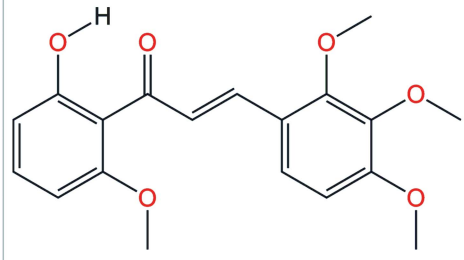
Structural data: full structural data are available from iucrdata.iucr.org

In the title molecule, $C_{19}H_{20}O_6$, the dihedral angle formed by the benzene rings is 36.71 (2)°. The hydroxy group is involved in a weak intramolecular O—H···O hydrogen bond. In the crystal, two weak C—H···O hydrogen bonds link the molecules into chains along [001].

3D view



Chemical scheme



Structure description

Chalcones are secondary metabolites found in plants with a C6–C3–C6 skeleton, a C3 skeleton being an α,β-unsaturated carbonyl (enone). A variety of chalcones have been isolated from natural sources and synthesized because they have shown wide spectrum of biological activities with clinical potentials against various diseases (Zhuang *et al.*, 2017). As part of our ongoing work in this area (Shin *et al.*, 2019; Lee *et al.*, 2016), the crystal structure of the title compound has been determined.

The molecular structure of the title compound is shown in Fig. 1. An intramolecular O5—H5···O1 hydrogen bond (Table 1) may cause the C1═O1 double bond [1.257 (5) Å] to be slightly longer than the normal value (Allen *et al.* 1987). The dihedral angle between the benzene rings is 36.71 (2)°. The *trans* configuration of the C2═C3 double bond is confirmed by the C1—C2═C3—C4 torsion angle of of −177.4 (4)°. The methoxy group on benzene ring *A*, which has a hydroxyl substituent, is almost coplanar with the ring plane [C17—C18—O6—C19 = −4.3 (5)°]. Of the three methoxy groups attached to benzene ring *B*, the two methoxy groups at the *ortho* and *meta* positions are significantly rotated from the ring plane [C4—C5—O2—C10 = 114.0 (4), C5—C6—O3—C11 = 113.9 (4)°] while the group in the *para* position is essentially coplanar [C6—C7—O4—C12 = −179.3 (3)°].

In the crystal, two weak C—H···O hydrogen bonds link the molecules into chains along [001] (Table 1, Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O1	0.84	1.76	2.502 (3)	147
C19—H19A···O1^{i}	0.98	2.53	3.490 (6)	167
C11—H11A···O3^{ii}	0.98	2.55	3.363 (5)	140

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

Synthesis and crystallization

To a solution of 2-hydroxy-6-methoxyacetophenone (166 mg, 1 mmol) in 15 ml of anhydrous ethanol was added 2,3,4-trimethoxybenzaldehyde (196 mg, 1 mmol) and the temperature was adjusted to around 276–277 K in an ice-bath. To the cooled reaction mixture was added 1.5 ml of 50% aqueous KOH solution, and the reaction mixture was stirred at room temperature for 24 h. After completion of the reaction (monitored by TLC), this mixture was poured into iced water (40 ml) and was acidified with a 6 *N* HCl solution until the pH was equal to 2 to produce a solid product. This solid was recrystallized from an ethanol solution to obtain single crystals of the title compound in 38% yield.

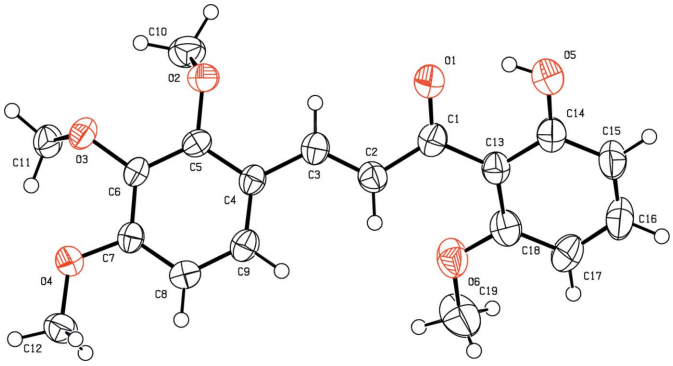


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme with displacement ellipsoids drawn at the 30% probability level.

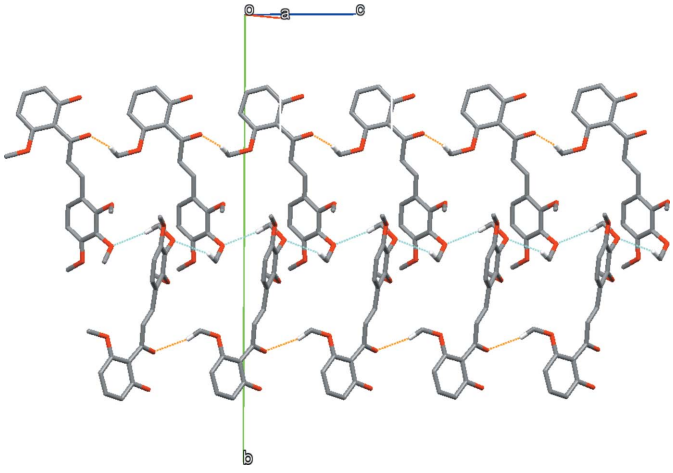


Figure 2
Part of the crystal structure with weak C19—H19A···O1^{i} and C11—H11A···O^{ii} hydrogen bonds shown as orange and blue dashed lines, respectively. For clarity only those H atoms involved in hydrogen bonding are shown.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{20}O_6$
M_r	344.35
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	200
a, b, c (Å)	7.7143 (5), 28.907 (2), 7.5010 (5)
V (Å^3)	1672.72 (19)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.18 × 0.09 × 0.08
Data collection	
Diffractometer	Bruker APEXII CCD area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11890, 4004, 2199
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.105, 0.95
No. of reflections	4004
No. of parameters	231
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.24

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008), *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bruker (2012). *APEX2*, *SAINT* and *SADABS*, Bruker AXS Inc. Madison, Wisconsin, USA.
Lee, D., Jung Jung, Y., Koh, D., Lim, Y., Lee, Y. & Shin, S. (2016). *Cancer Lett.* **372**, 1–9.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Shin, S., Lee, J., Park, J., Lee, Y., Ahn, S., Lee, J., Koh, D., Lee, Y. & Lim, Y. (2019). *Bioorg. Chem.* **83**, 438–449.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zhuang, C., Zhang, W., Sheng, C., Zhang, W., Xing, C. & Miao, Z. (2017). *Chem. Rev.* **117**, 7762–7810.

full crystallographic data

IUCrData (2019). **4**, x190179 [https://doi.org/10.1107/S2414314619001792]

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

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Crystal data

$C_{19}H_{20}O_6$
M_r = 344.35
Orthorhombic, $Pna2_1$
a = 7.7143 (5) Å
b = 28.907 (2) Å
c = 7.5010 (5) Å
V = 1672.72 (19) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.367 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3178 reflections
θ = 2.7–27.3°
μ = 0.10 mm^{-1}
T = 200 K
Block, yellow
0.18 × 0.09 × 0.08 mm

Data collection

Bruker APEXII CCD area detector diffractometer
/f and /w scans
11890 measured reflections
4004 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 28.3°, θ_{min} = 2.7°
h = −10→10
k = −38→29
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.105
S = 0.95
4004 reflections
231 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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	0.8643 (4)	0.25927 (8)	0.3218 (4)	0.0524 (7)
C1	0.7768 (5)	0.27219 (13)	0.1895 (6)	0.0411 (9)

C2	0.7215 (5)	0.32059 (13)	0.1845 (5)	0.0418 (10)
H2	0.6322	0.3295	0.1045	0.050*
C3	0.7932 (4)	0.35234 (12)	0.2894 (5)	0.0405 (10)
H3	0.8853	0.3422	0.3638	0.049*
C4	0.7459 (4)	0.40117 (12)	0.3028 (5)	0.0350 (8)
C5	0.8519 (4)	0.43163 (12)	0.3987 (5)	0.0352 (9)
C6	0.8156 (4)	0.47884 (12)	0.4073 (5)	0.0343 (9)
C7	0.6691 (4)	0.49594 (11)	0.3223 (5)	0.0351 (9)
C8	0.5596 (5)	0.46635 (12)	0.2301 (5)	0.0401 (10)
H8	0.4590	0.4780	0.1725	0.048*
C9	0.5984 (5)	0.41971 (12)	0.2229 (5)	0.0411 (10)
H9	0.5218	0.3996	0.1613	0.049*
O2	0.9906 (3)	0.41348 (8)	0.4897 (4)	0.0460 (7)
C10	1.1582 (5)	0.42676 (14)	0.4274 (6)	0.0555 (12)
H10A	1.1545	0.4312	0.2979	0.083*
H10B	1.2422	0.4025	0.4564	0.083*
H10C	1.1930	0.4557	0.4850	0.083*
O3	0.9171 (3)	0.50681 (8)	0.5123 (4)	0.0419 (6)
C11	1.0189 (5)	0.54028 (12)	0.4170 (6)	0.0446 (10)
H11A	1.0929	0.5244	0.3302	0.067*
H11B	1.0915	0.5575	0.5013	0.067*
H11C	0.9418	0.5618	0.3546	0.067*
O4	0.6431 (3)	0.54259 (8)	0.3408 (4)	0.0414 (6)
C12	0.4934 (5)	0.56240 (13)	0.2583 (6)	0.0484 (10)
H12A	0.4955	0.5558	0.1302	0.073*
H12B	0.4936	0.5960	0.2770	0.073*
H12C	0.3886	0.5490	0.3112	0.073*
C13	0.7259 (5)	0.23817 (13)	0.0556 (5)	0.0400 (10)
C14	0.7381 (5)	0.19086 (14)	0.0973 (6)	0.0466 (11)
O5	0.7991 (4)	0.17618 (8)	0.2566 (5)	0.0603 (9)
H5	0.8340	0.1990	0.3160	0.090*
C15	0.6852 (5)	0.15690 (14)	−0.0206 (7)	0.0594 (13)
H15	0.6941	0.1252	0.0111	0.071*
C16	0.6202 (6)	0.16929 (15)	−0.1831 (8)	0.0672 (13)
H16	0.5820	0.1459	−0.2628	0.081*
C17	0.6087 (5)	0.21516 (15)	−0.2341 (6)	0.0584 (12)
H17	0.5643	0.2232	−0.3481	0.070*
C18	0.6626 (5)	0.24917 (14)	−0.1170 (6)	0.0481 (11)
O6	0.6613 (4)	0.29485 (9)	−0.1609 (4)	0.0586 (8)
C19	0.6133 (8)	0.30746 (16)	−0.3385 (7)	0.0826 (17)
H19A	0.6803	0.2890	−0.4238	0.124*
H19B	0.6375	0.3404	−0.3574	0.124*
H19C	0.4894	0.3016	−0.3560	0.124*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0626 (18)	0.0431 (16)	0.0514 (18)	0.0019 (13)	−0.0090 (16)	−0.0047 (15)

C1	0.039 (2)	0.042 (2)	0.042 (2)	−0.0028 (18)	0.006 (2)	−0.006 (2)
C2	0.047 (2)	0.039 (2)	0.040 (2)	0.0012 (18)	0.001 (2)	−0.001 (2)
C3	0.042 (2)	0.037 (2)	0.043 (3)	−0.0040 (17)	0.003 (2)	−0.0014 (19)
C4	0.036 (2)	0.036 (2)	0.033 (2)	−0.0061 (16)	0.0016 (18)	−0.0017 (17)
C5	0.037 (2)	0.041 (2)	0.028 (2)	−0.0014 (17)	−0.0005 (17)	−0.0006 (18)
C6	0.037 (2)	0.036 (2)	0.030 (2)	−0.0041 (16)	−0.0009 (18)	−0.0071 (18)
C7	0.038 (2)	0.035 (2)	0.032 (2)	−0.0035 (16)	0.0050 (19)	−0.0024 (19)
C8	0.036 (2)	0.046 (2)	0.038 (2)	−0.0007 (18)	−0.0035 (19)	0.001 (2)
C9	0.038 (2)	0.042 (2)	0.043 (3)	−0.0083 (17)	0.002 (2)	−0.0042 (19)
O2	0.0443 (16)	0.0426 (16)	0.0510 (17)	−0.0010 (12)	−0.0114 (15)	0.0046 (14)
C10	0.042 (2)	0.068 (3)	0.056 (3)	0.007 (2)	−0.005 (2)	−0.005 (2)
O3	0.0495 (15)	0.0412 (15)	0.0350 (16)	−0.0090 (13)	−0.0047 (13)	−0.0037 (13)
C11	0.048 (2)	0.037 (2)	0.049 (3)	−0.0103 (18)	−0.001 (2)	0.002 (2)
O4	0.0450 (15)	0.0356 (14)	0.0435 (16)	0.0039 (11)	−0.0025 (14)	−0.0022 (13)
C12	0.047 (2)	0.049 (2)	0.049 (3)	0.010 (2)	−0.004 (2)	0.001 (2)
C13	0.040 (2)	0.037 (2)	0.044 (3)	−0.0010 (17)	0.0049 (19)	−0.003 (2)
C14	0.047 (3)	0.042 (3)	0.051 (3)	−0.003 (2)	0.002 (2)	−0.006 (2)
O5	0.070 (2)	0.0420 (17)	0.069 (2)	−0.0001 (15)	−0.0086 (18)	0.0004 (17)
C15	0.066 (3)	0.035 (2)	0.077 (4)	−0.005 (2)	−0.003 (3)	−0.010 (3)
C16	0.079 (3)	0.047 (3)	0.076 (4)	−0.007 (2)	−0.007 (3)	−0.022 (3)
C17	0.072 (3)	0.058 (3)	0.046 (3)	−0.002 (2)	−0.005 (2)	−0.016 (2)
C18	0.052 (3)	0.042 (2)	0.050 (3)	0.001 (2)	0.011 (2)	−0.004 (2)
O6	0.088 (2)	0.0452 (18)	0.0424 (18)	0.0033 (15)	0.0042 (18)	−0.0045 (15)
C19	0.148 (5)	0.067 (3)	0.033 (3)	0.025 (3)	0.003 (3)	0.010 (3)

Geometric parameters (Å, º)

O1—O1	0.000 (8)	O3—C11	1.437 (4)
O1—C1	1.257 (5)	C11—H11A	0.9800
C1—O1	1.257 (5)	C11—H11B	0.9800
C1—C13	1.460 (5)	C11—H11C	0.9800
C1—C2	1.463 (5)	O4—C12	1.430 (4)
C2—C3	1.329 (5)	C12—H12A	0.9800
C2—H2	0.9500	C12—H12B	0.9800
C3—C4	1.462 (5)	C12—H12C	0.9800
C3—H3	0.9500	C13—C14	1.406 (5)
C4—C9	1.393 (5)	C13—C18	1.420 (6)
C4—C5	1.401 (5)	C14—O5	1.353 (5)
C5—O2	1.373 (4)	C14—C15	1.383 (5)
C5—C6	1.395 (5)	O5—H5	0.8400
C6—O3	1.374 (4)	C15—C16	1.366 (6)
C6—C7	1.388 (5)	C15—H15	0.9500
C7—O4	1.370 (4)	C16—C17	1.383 (6)
C7—C8	1.387 (5)	C16—H16	0.9500
C8—C9	1.382 (4)	C17—C18	1.382 (5)
C8—H8	0.9500	C17—H17	0.9500
C9—H9	0.9500	C18—O6	1.361 (5)
O2—C10	1.428 (4)	O6—C19	1.430 (5)

C10—H10A	0.9800	C19—H19A	0.9800
C10—H10B	0.9800	C19—H19B	0.9800
C10—H10C	0.9800	C19—H19C	0.9800
O1—C1—C13	119.2 (4)	O3—C11—H11B	109.5
O1—C1—C13	119.2 (4)	H11A—C11—H11B	109.5
O1—C1—C2	117.4 (4)	O3—C11—H11C	109.5
O1—C1—C2	117.4 (4)	H11A—C11—H11C	109.5
C13—C1—C2	123.2 (4)	H11B—C11—H11C	109.5
C3—C2—C1	121.6 (4)	C7—O4—C12	117.9 (3)
C3—C2—H2	119.2	O4—C12—H12A	109.5
C1—C2—H2	119.2	O4—C12—H12B	109.5
C2—C3—C4	127.1 (4)	H12A—C12—H12B	109.5
C2—C3—H3	116.4	O4—C12—H12C	109.5
C4—C3—H3	116.4	H12A—C12—H12C	109.5
C9—C4—C5	117.1 (3)	H12B—C12—H12C	109.5
C9—C4—C3	123.1 (3)	C14—C13—C18	116.4 (4)
C5—C4—C3	119.8 (3)	C14—C13—C1	118.9 (4)
O2—C5—C6	120.5 (3)	C18—C13—C1	124.7 (4)
O2—C5—C4	118.0 (3)	O5—C14—C15	116.4 (4)
C6—C5—C4	121.4 (3)	O5—C14—C13	121.7 (4)
O3—C6—C7	121.2 (3)	C15—C14—C13	121.9 (4)
O3—C6—C5	119.2 (3)	C14—O5—H5	109.5
C7—C6—C5	119.4 (3)	C16—C15—C14	119.5 (4)
O4—C7—C8	124.6 (3)	C16—C15—H15	120.2
O4—C7—C6	115.0 (3)	C14—C15—H15	120.2
C8—C7—C6	120.3 (3)	C15—C16—C17	121.4 (4)
C9—C8—C7	119.3 (3)	C15—C16—H16	119.3
C9—C8—H8	120.4	C17—C16—H16	119.3
C7—C8—H8	120.4	C18—C17—C16	119.1 (4)
C8—C9—C4	122.4 (3)	C18—C17—H17	120.4
C8—C9—H9	118.8	C16—C17—H17	120.4
C4—C9—H9	118.8	O6—C18—C17	122.3 (4)
C5—O2—C10	116.1 (3)	O6—C18—C13	116.1 (4)
O2—C10—H10A	109.5	C17—C18—C13	121.6 (4)
O2—C10—H10B	109.5	C18—O6—C19	118.3 (3)
H10A—C10—H10B	109.5	O6—C19—H19A	109.5
O2—C10—H10C	109.5	O6—C19—H19B	109.5
H10A—C10—H10C	109.5	H19A—C19—H19B	109.5
H10B—C10—H10C	109.5	O6—C19—H19C	109.5
C6—O3—C11	115.0 (3)	H19A—C19—H19C	109.5
O3—C11—H11A	109.5	H19B—C19—H19C	109.5
O1—O1—C1—C13	0.0 (4)	C7—C6—O3—C11	−71.8 (4)
O1—O1—C1—C2	0.0 (6)	C5—C6—O3—C11	113.9 (4)
O1—C1—C2—C3	16.0 (6)	C8—C7—O4—C12	−0.5 (5)
O1—C1—C2—C3	16.0 (6)	C6—C7—O4—C12	−179.3 (3)
C13—C1—C2—C3	−168.1 (3)	O1—C1—C13—C14	15.3 (5)

C1—C2—C3—C4	−177.4 (4)	O1—C1—C13—C14	15.3 (5)
C2—C3—C4—C9	9.7 (6)	C2—C1—C13—C14	−160.5 (4)
C2—C3—C4—C5	−169.9 (4)	O1—C1—C13—C18	−165.1 (4)
C9—C4—C5—O2	175.1 (3)	O1—C1—C13—C18	−165.1 (4)
C3—C4—C5—O2	−5.2 (5)	C2—C1—C13—C18	19.1 (6)
C9—C4—C5—C6	−2.8 (5)	C18—C13—C14—O5	178.9 (4)
C3—C4—C5—C6	176.9 (3)	C1—C13—C14—O5	−1.4 (6)
O2—C5—C6—O3	−2.0 (5)	C18—C13—C14—C15	−2.4 (6)
C4—C5—C6—O3	175.9 (3)	C1—C13—C14—C15	177.3 (4)
O2—C5—C6—C7	−176.4 (3)	O5—C14—C15—C16	179.2 (4)
C4—C5—C6—C7	1.4 (5)	C13—C14—C15—C16	0.4 (6)
O3—C6—C7—O4	4.7 (5)	C14—C15—C16—C17	1.2 (7)
C5—C6—C7—O4	179.0 (3)	C15—C16—C17—C18	−0.7 (7)
O3—C6—C7—C8	−174.1 (3)	C16—C17—C18—O6	177.4 (4)
C5—C6—C7—C8	0.2 (5)	C16—C17—C18—C13	−1.4 (6)
O4—C7—C8—C9	−179.1 (3)	C14—C13—C18—O6	−176.0 (3)
C6—C7—C8—C9	−0.4 (5)	C1—C13—C18—O6	4.4 (6)
C7—C8—C9—C4	−1.1 (6)	C14—C13—C18—C17	2.9 (6)
C5—C4—C9—C8	2.6 (5)	C1—C13—C18—C17	−176.8 (4)
C3—C4—C9—C8	−177.0 (4)	C17—C18—O6—C19	−4.3 (6)
C6—C5—O2—C10	−68.1 (4)	C13—C18—O6—C19	174.6 (4)
C4—C5—O2—C10	114.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19*A*···O1^{i}	0.98	2.53	3.490 (6)	167
C11—H11*A*···O3^{ii}	0.98	2.55	3.363 (5)	140
O5—H5···O1	0.84	1.76	2.502 (3)	147

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