Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(3,5-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

Seunghyun Ahn,[a] Ha-Jin Lee,[b] Yoongho Lim[c] and Dongsoo Koh[a]*

[a]Department of Applied Chemistry, Dongduk Women's University, Seoul 136-714, Republic of Korea, [b]Jeonju Center, Korea Basic Science Center (KBSI), Jeonju 561-765, Republic of Korea, and [c]Division of Bioscience and Biotechnology, BMIC, Konkuk University, Seoul 143-701, Republic of Korea
Correspondence e-mail: dskoh@dongduk.ac.kr

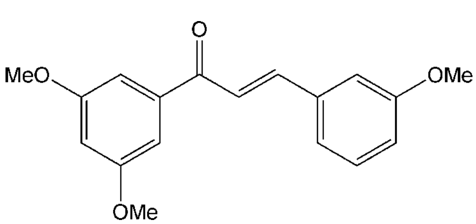
Received 22 February 2013; accepted 2 April 2013

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.115; data-to-parameter ratio = 13.0.

In the title molecule, $C_{18}H_{18}O_4$, the C=C bond of the central enone group adopts a *trans* conformation. The relative conformation of the C=O and C=C bonds is *s-cisoid*. The dihedral angle between the planes of the benzene rings is 29.49 (12)°. In the crystal, weak C—H⋯O hydrogen bonds link the molecules into chains along [010].

Related literature

For the synthesis and biological properties of chalcone derivatives, see: Shenvi *et al.* (2013); Hsieh *et al.* (2012); Hwang *et al.* (2011); Jo *et al.* (2012); Sharma *et al.* (2012); Sashidhara *et al.* (2011). For related structures, see: Carvalho-Jr *et al.* (2011); Wu *et al.* (2012).



Experimental

Crystal data

$C_{18}H_{18}O_4$
M_r = 298.32
Triclinic, $P\bar{1}$
a = 8.2402 (12) Å
b = 9.1449 (14) Å
c = 10.8876 (16) Å
α = 95.395 (3)°
β = 107.667 (3)°
γ = 102.837 (3)°
V = 750.61 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 200 K
0.33 × 0.26 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.970, T_{max} = 0.989
4383 measured reflections
2619 independent reflections
1531 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 0.98
2619 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7⋯O4^{i}	0.95	2.55	3.405 (3)	151
C9—H9C⋯O1^{ii}	0.98	2.50	3.388 (3)	150

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5590).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carvalho-Jr, P. S., Sallum, L. O., Cidade, A. F., Aquino, G. L. B. & Napolitano, H. B. (2011). *Acta Cryst.* E**67**, o2126.
Hsieh, C.-T., Hsieh, T.-J., El-Shazly, M., Chuang, D.-W., Tsai, Y.-H., Yen a, C.-T., Wua, S.-F., Wua, Y.-C. & Chang, F.-R. (2012). *Bioorg. Med. Chem. Lett.* **22**, 3912–3915.
Hwang, D., Hyun, J., Jo, G., Koh, D. & Lim, Y. (2011). *Magn. Reson. Chem.* **49**, 41–45.
Jo, G., Sung, S. H., Lee, Y., Kim, B.-G., Yoon, J. W., Lee, H. K., Ji, S. Y., Koh, D., Ahn, J. H. & Lim, Y. (2012). *Bull. Korean Chem. Soc.* **33**, 3841–3844.
Sashidhara, K. V., Kumar, M., Modukuri, R. M., Sonkar, R., Bhatia, G., Khanna, A. K., Rai, S. V. & Shukla, R. (2011). *Bioorg. Med. Chem. Lett.* **21**, 4480–4484.
Sharma, V., Singh, G., Kaur, H., Saxena, A. K. & Ishar, M. P. S. (2012). *Bioorg. Med. Chem. Lett.* **22**, 6343–6346.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shenvi, S., Kumar, K., Hatti, K. S., Rijesh, K., Diwakar, L. & Reddy, G. C. (2013). *Eur. J. Med. Chem.* **62**, 435–442.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wu, J., Qiu, J., Wu, X., Yang, S. & Liu, Y. (2012). *Acta Cryst.* E**68**, o154.

supplementary materials

Acta Cryst. (2013). E**69**, o666 [doi:10.1107/S1600536813008982]

(*E*)-1-(3,5-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

Seunghyun Ahn, Ha-Jin Lee, Yoongho Lim and Dongsoo Koh

Comment

Chalcones are one of the secondary metabolites in plants and belong to a flavonoid class. They have shown diverse biological activities including anti-cancer (Shenvi *et al.* 2013), anti-microbial (Sharma *et al.* 2012), anti-diabetic (Hsieh *et al.* 2012) and anti-inflammatory (Sashidhara *et al.* 2011). As a part of our studies on the substituent effects of chalcones on structures and biological activities (Jo *et al.*, 2012; Hwang *et al.*, 2011), the title compound was synthesized and its crystal structure was determined.

The molecular structure of the title compound is shown in Fig. 1. The *trans* configuration of C2═C3 double bond is defined by the dihedral angle of -177.9 (2) Å for C1—C2—C3—C4. The relative conformation of two double bonds, O1═C1 and C2═C3, is s-*cisoid* with a torsion angle of -12.8 (4)° for O1—C1—C2—C3. The orientations of the three methoxy groups can be defined by the torsion angles C9—O2—C8—C10 [177.1 (3)°], C17—O4—C16—C15 [-1.8 (5)°] and C14—O3—C13—C15 [-172.2 (3)°]. The dihedral angle between the benzene rings is 29.49 (12)°. In the crystal, weak C—H···O hydrogen bonds links the molecules into chains along [010] (Fig. 2). Some examples of methoxy substituted chalcone structures have been published (Wu *et al.*, 2012; Carvalho-Jr *et al.*, 2011).

Experimental

To a solution of 3-methoxybenzaldehyde (136 mg, 1 mmol) in 20 ml of ethanol was added 3,5-dimethoxyacetophenone (180 mg, 1 mmol) and the temperature was adjusted to around 276 K in an ice-bath. To the cooled reaction mixture was added 1 ml of 50% aqueous KOH solution, and the reaction mixture was stirred at room temperature for 20 h. This mixture was poured into iced water (30 ml) was acidified (pH =3) with 3 N HCl solution to give a precipitate. Filtration and washing with water afforded crude solid of the title compound (240 mg, 79%). Recrystallization of the solid in ethanol gave pale yellow crystals which were suitable for X-ray diffraction (mp: 363–364 K).

Refinement

H atoms were placed in calculated positions and refined as riding with C—H = 0.95–0.98 Å, and U_{iso}(H) = 1.2 U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}). Four outliers were removed in the final refinement (4 2 8, -7 5 9, -7, -3, 10 and -7, -4, 9).

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

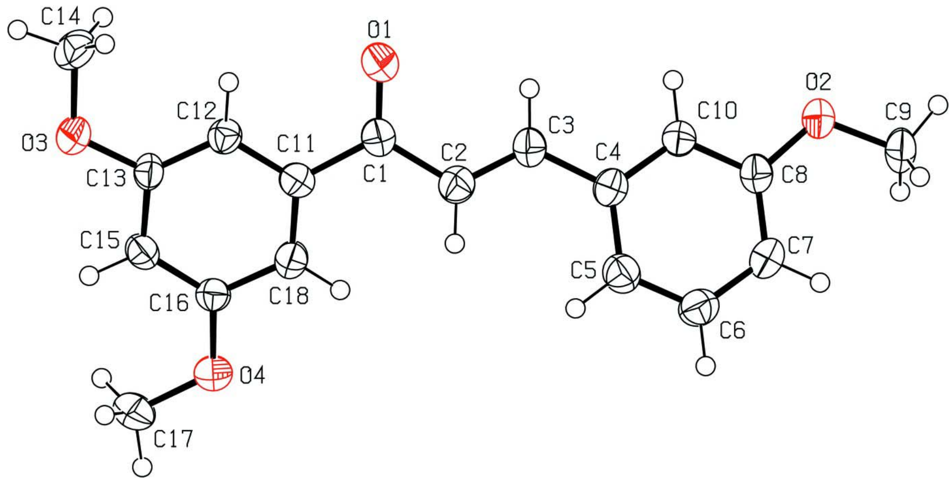


Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

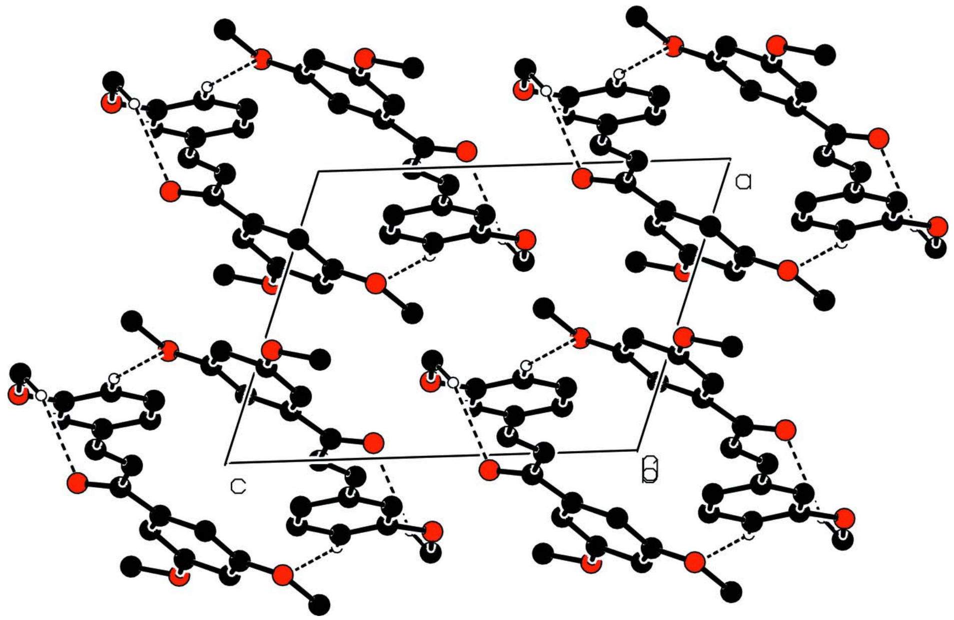


Figure 2

Part of the crystal structure with weak intermolecular C—H···O hydrogen bonds shown as dashed lines.

(*E*)-1-(3,5-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{18}O_4$	γ = 102.837 (3)°
M_r = 298.32	V = 750.61 (19) Å^3
Triclinic, $P\overline{1}$	Z = 2
Hall symbol: -P 1	F(000) = 316
a = 8.2402 (12) Å	D_x = 1.320 Mg m^{-3}
b = 9.1449 (14) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
c = 10.8876 (16) Å	Cell parameters from 1553 reflections
α = 95.395 (3)°	θ = 2.3–27.9°
β = 107.667 (3)°	μ = 0.09 mm^{-1}

T = 200 K
Block, pale yellow

0.33 × 0.26 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.970, T_{max} = 0.989

4383 measured reflections
2619 independent reflections
1531 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −7→9
k = −9→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 0.98
2619 reflections
202 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.0722 (3)	0.0058 (3)	0.7610 (2)	0.0354 (6)
O1	0.0527 (2)	−0.06317 (18)	0.65288 (16)	0.0503 (5)
C2	−0.0008 (3)	0.1386 (3)	0.7724 (2)	0.0385 (6)
H2	−0.0059	0.1771	0.8549	0.046*
C3	−0.0595 (3)	0.2054 (3)	0.6708 (2)	0.0355 (6)
H3	−0.0560	0.1615	0.5893	0.043*
C4	−0.1290 (3)	0.3399 (2)	0.6711 (2)	0.0338 (6)
C5	−0.1565 (3)	0.4081 (3)	0.7811 (2)	0.0413 (6)
H5	−0.1313	0.3665	0.8594	0.050*
C6	−0.2199 (3)	0.5353 (3)	0.7760 (2)	0.0435 (7)
H6	−0.2397	0.5798	0.8508	0.052*
C7	−0.2555 (3)	0.6000 (3)	0.6635 (2)	0.0407 (6)
H7	−0.2990	0.6879	0.6611	0.049*
C8	−0.2268 (3)	0.5343 (3)	0.5553 (2)	0.0359 (6)
O2	−0.2555 (2)	0.58787 (18)	0.43929 (15)	0.0456 (5)

C9	−0.3268 (3)	0.7168 (3)	0.4271 (2)	0.0462 (7)
H9A	−0.4436	0.6916	0.4371	0.069*
H9B	−0.3375	0.7444	0.3407	0.069*
H9C	−0.2481	0.8028	0.4952	0.069*
C10	−0.1656 (3)	0.4048 (2)	0.5588 (2)	0.0341 (6)
H10	−0.1485	0.3597	0.4831	0.041*
C11	0.1748 (3)	−0.0419 (3)	0.8812 (2)	0.0325 (6)
C12	0.2212 (3)	−0.1790 (2)	0.8674 (2)	0.0323 (6)
H12	0.1800	−0.2429	0.7842	0.039*
C13	0.3267 (3)	−0.2205 (2)	0.9747 (2)	0.0329 (6)
O3	0.3825 (2)	−0.35105 (17)	0.97449 (15)	0.0466 (5)
C14	0.3448 (4)	−0.4415 (3)	0.8493 (2)	0.0493 (7)
H14A	0.2167	−0.4794	0.8067	0.074*
H14B	0.3970	−0.5278	0.8611	0.074*
H14C	0.3951	−0.3791	0.7947	0.074*
C15	0.3874 (3)	−0.1279 (2)	1.0980 (2)	0.0342 (6)
H15	0.4604	−0.1577	1.1718	0.041*
C16	0.3407 (3)	0.0062 (3)	1.1115 (2)	0.0321 (6)
O4	0.3951 (2)	0.10519 (17)	1.22797 (15)	0.0441 (5)
C17	0.5018 (4)	0.0614 (3)	1.3408 (2)	0.0491 (7)
H17A	0.4375	−0.0362	1.3542	0.074*
H17B	0.5295	0.1393	1.4177	0.074*
H17C	0.6116	0.0510	1.3279	0.074*
C18	0.2335 (3)	0.0503 (3)	1.0033 (2)	0.0349 (6)
H18	0.2008	0.1428	1.0131	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0339 (15)	0.0363 (14)	0.0369 (15)	0.0102 (12)	0.0112 (13)	0.0105 (12)
O1	0.0648 (14)	0.0511 (11)	0.0357 (11)	0.0269 (10)	0.0094 (10)	0.0079 (9)
C2	0.0385 (16)	0.0407 (15)	0.0398 (15)	0.0142 (13)	0.0146 (13)	0.0088 (12)
C3	0.0323 (15)	0.0366 (14)	0.0396 (15)	0.0115 (12)	0.0115 (13)	0.0117 (11)
C4	0.0272 (14)	0.0336 (14)	0.0406 (15)	0.0078 (11)	0.0114 (12)	0.0069 (11)
C5	0.0418 (16)	0.0444 (15)	0.0401 (15)	0.0131 (13)	0.0146 (14)	0.0118 (12)
C6	0.0485 (18)	0.0438 (16)	0.0443 (16)	0.0163 (14)	0.0213 (14)	0.0069 (13)
C7	0.0427 (17)	0.0354 (15)	0.0482 (16)	0.0174 (13)	0.0158 (14)	0.0078 (12)
C8	0.0330 (15)	0.0375 (15)	0.0388 (15)	0.0129 (12)	0.0102 (13)	0.0113 (12)
O2	0.0589 (13)	0.0466 (11)	0.0424 (11)	0.0297 (9)	0.0186 (10)	0.0166 (8)
C9	0.0516 (18)	0.0417 (16)	0.0501 (17)	0.0249 (14)	0.0123 (15)	0.0168 (13)
C10	0.0353 (15)	0.0354 (14)	0.0342 (14)	0.0126 (12)	0.0123 (12)	0.0081 (11)
C11	0.0292 (14)	0.0365 (14)	0.0313 (14)	0.0071 (11)	0.0101 (12)	0.0063 (11)
C12	0.0325 (14)	0.0287 (13)	0.0342 (14)	0.0077 (11)	0.0096 (12)	0.0040 (11)
C13	0.0361 (15)	0.0306 (13)	0.0360 (14)	0.0148 (12)	0.0117 (12)	0.0110 (11)
O3	0.0637 (13)	0.0386 (10)	0.0405 (10)	0.0267 (10)	0.0127 (10)	0.0056 (8)
C14	0.0572 (19)	0.0384 (15)	0.0497 (17)	0.0190 (14)	0.0129 (15)	−0.0035 (13)
C15	0.0348 (15)	0.0396 (15)	0.0300 (14)	0.0115 (12)	0.0107 (12)	0.0112 (11)
C16	0.0325 (14)	0.0352 (14)	0.0297 (13)	0.0101 (12)	0.0110 (12)	0.0051 (11)
O4	0.0544 (12)	0.0433 (10)	0.0340 (10)	0.0195 (9)	0.0105 (9)	0.0016 (8)
C17	0.0529 (18)	0.0585 (18)	0.0314 (15)	0.0178 (15)	0.0069 (14)	0.0031 (13)

C18	0.0347 (15)	0.0342 (14)	0.0412 (15)	0.0135 (12)	0.0158 (13)	0.0113 (12)

Geometric parameters (Å, º)

C1—O1	1.229 (2)	C10—H10	0.9500
C1—C2	1.481 (3)	C11—C18	1.390 (3)
C1—C11	1.489 (3)	C11—C12	1.397 (3)
C2—C3	1.329 (3)	C12—C13	1.371 (3)
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.467 (3)	C13—O3	1.371 (2)
C3—H3	0.9500	C13—C15	1.400 (3)
C4—C10	1.389 (3)	O3—C14	1.433 (2)
C4—C5	1.401 (3)	C14—H14A	0.9800
C5—C6	1.376 (3)	C14—H14B	0.9800
C5—H5	0.9500	C14—H14C	0.9800
C6—C7	1.390 (3)	C15—C16	1.373 (3)
C6—H6	0.9500	C15—H15	0.9500
C7—C8	1.379 (3)	C16—O4	1.373 (2)
C7—H7	0.9500	C16—C18	1.395 (3)
C8—O2	1.370 (2)	O4—C17	1.426 (3)
C8—C10	1.385 (3)	C17—H17A	0.9800
O2—C9	1.429 (2)	C17—H17B	0.9800
C9—H9A	0.9800	C17—H17C	0.9800
C9—H9B	0.9800	C18—H18	0.9500
C9—H9C	0.9800		
O1—C1—C2	120.5 (2)	C4—C10—H10	119.4
O1—C1—C11	119.6 (2)	C18—C11—C12	120.0 (2)
C2—C1—C11	119.9 (2)	C18—C11—C1	121.7 (2)
C3—C2—C1	122.0 (2)	C12—C11—C1	118.1 (2)
C3—C2—H2	119.0	C13—C12—C11	119.5 (2)
C1—C2—H2	119.0	C13—C12—H12	120.2
C2—C3—C4	126.9 (2)	C11—C12—H12	120.2
C2—C3—H3	116.6	C12—C13—O3	125.4 (2)
C4—C3—H3	116.6	C12—C13—C15	120.8 (2)
C10—C4—C5	118.3 (2)	O3—C13—C15	113.8 (2)
C10—C4—C3	118.9 (2)	C13—O3—C14	116.83 (18)
C5—C4—C3	122.7 (2)	O3—C14—H14A	109.5
C6—C5—C4	120.1 (2)	O3—C14—H14B	109.5
C6—C5—H5	120.0	H14A—C14—H14B	109.5
C4—C5—H5	120.0	O3—C14—H14C	109.5
C5—C6—C7	121.2 (2)	H14A—C14—H14C	109.5
C5—C6—H6	119.4	H14B—C14—H14C	109.5
C7—C6—H6	119.4	C16—C15—C13	119.6 (2)
C8—C7—C6	118.9 (2)	C16—C15—H15	120.2
C8—C7—H7	120.5	C13—C15—H15	120.2
C6—C7—H7	120.5	C15—C16—O4	123.8 (2)
O2—C8—C7	124.4 (2)	C15—C16—C18	120.3 (2)
O2—C8—C10	115.29 (19)	O4—C16—C18	115.8 (2)
C7—C8—C10	120.3 (2)	C16—O4—C17	117.02 (18)

C8—O2—C9	118.08 (17)	O4—C17—H17A	109.5
O2—C9—H9A	109.5	O4—C17—H17B	109.5
O2—C9—H9B	109.5	H17A—C17—H17B	109.5
H9A—C9—H9B	109.5	O4—C17—H17C	109.5
O2—C9—H9C	109.5	H17A—C17—H17C	109.5
H9A—C9—H9C	109.5	H17B—C17—H17C	109.5
H9B—C9—H9C	109.5	C11—C18—C16	119.7 (2)
C8—C10—C4	121.2 (2)	C11—C18—H18	120.2
C8—C10—H10	119.4	C16—C18—H18	120.2
O1—C1—C2—C3	−12.8 (4)	O1—C1—C11—C12	−10.3 (3)
C11—C1—C2—C3	165.3 (2)	C2—C1—C11—C12	171.6 (2)
C1—C2—C3—C4	−177.9 (2)	C18—C11—C12—C13	−0.8 (3)
C2—C3—C4—C10	172.7 (2)	C1—C11—C12—C13	175.2 (2)
C2—C3—C4—C5	−5.9 (4)	C11—C12—C13—O3	−179.6 (2)
C10—C4—C5—C6	0.6 (4)	C11—C12—C13—C15	0.4 (3)
C3—C4—C5—C6	179.2 (2)	C12—C13—O3—C14	7.9 (3)
C4—C5—C6—C7	−0.9 (4)	C15—C13—O3—C14	−172.10 (19)
C5—C6—C7—C8	0.2 (4)	C12—C13—C15—C16	0.0 (3)
C6—C7—C8—O2	−179.5 (2)	O3—C13—C15—C16	−180.0 (2)
C6—C7—C8—C10	0.9 (4)	C13—C15—C16—O4	−179.33 (19)
C7—C8—O2—C9	−2.6 (4)	C13—C15—C16—C18	0.0 (3)
C10—C8—O2—C9	177.0 (2)	C15—C16—O4—C17	−2.0 (3)
O2—C8—C10—C4	179.2 (2)	C18—C16—O4—C17	178.7 (2)
C7—C8—C10—C4	−1.2 (4)	C12—C11—C18—C16	0.8 (3)
C5—C4—C10—C8	0.4 (4)	C1—C11—C18—C16	−175.1 (2)
C3—C4—C10—C8	−178.2 (2)	C15—C16—C18—C11	−0.4 (3)
O1—C1—C11—C18	165.7 (2)	O4—C16—C18—C11	178.96 (19)
C2—C1—C11—C18	−12.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O4^{i}	0.95	2.55	3.405 (3)	151
C9—H9*C*···O1^{ii}	0.98	2.50	3.388 (3)	150

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