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(*E*)-1-(2-Hydroxy-6-methoxyphenyl)-3-(2-methoxynaphthalen-1-yl)prop-2-en-1-one

Jiha Sung*

Department of Applied Chemistry, Dongduk Women's University, Seoul 136-714, Republic of Korea. *Correspondence e-mail: dddklab@gmail.com

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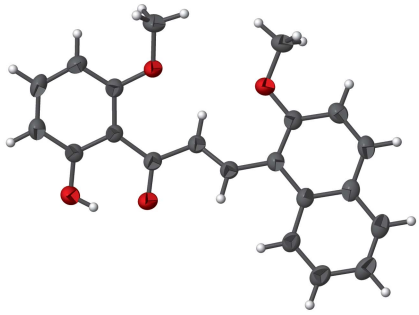
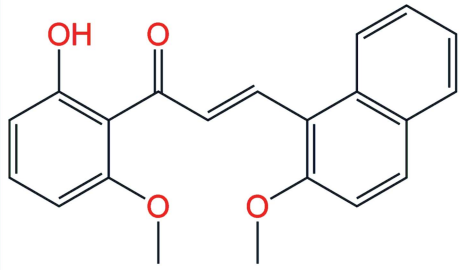
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{21}H_{18}O_4$, the dihedral angle between the naphthelene ring system (r.m.s. deviation = 0.014 Å) and the benzene ring is 9.68 (1)°. The C atom of the methoxy group of the naphthalene ring system is almost coplanar with the ring [C—O—C—C = −2.0 (3)°], whereas the C atom of the methoxy group of the phenol ring is slightly twisted [C—O—C—C = 6.2 (3)°]. An intramolecular O—H···O hydrogen bond generates an *S*(6) ring motif.

3D view



Chemical scheme



Structure description

Chalcones are a family of flavonoids which have a general C6–C3–C6 carbon framework. In contrast to other flavonoid families, chalcones possess the C3 unit as an α, β-unsaturated carbonyl (enone) group. In the enone system, the C═C and C═O double bonds are in a *cis* conformation along with a single bond connecting the two double bonds (*s-cisoid*). According to recent reviews (Mahapatra *et al.*, 2019; Zhuang *et al.*, 2017), natural and synthetic chalcones reveal diverse biological activities. In addition, when a chalcone has a hydroxyl group adjacent to carbonyl group, it tends to react *via* an intramolecular Michael addition to form flavanone, flavone or flavonol derivatives depending on the reaction conditions. The crystal structure of a flavone derived from hydroxyl-chalcone was described recently (Sung, 2018). In continuation of our work in this area (Ahn *et al.*, 2017), the title compound was synthesized and its crystal structure was determined and are reported here.

The molecular structure of the title compound is shown in Fig. 1. The C2═C3 double bond of the central enone group adopts a *trans* configuration [H2—C2—C3—H3 = −179.2°]. In the enone system, the C=C double bond and C=O double bond are not in the same plane [torsion angle O1—C1—C2—C3 = −17.6 (3)°]. The C atom of the methoxy group of the naphthalene ring is almost coplanar with the ring [C14—O2—C5—C6 = −2.0 (3)°], whereas the C atom of the methoxy group of the benzene ring is slightly

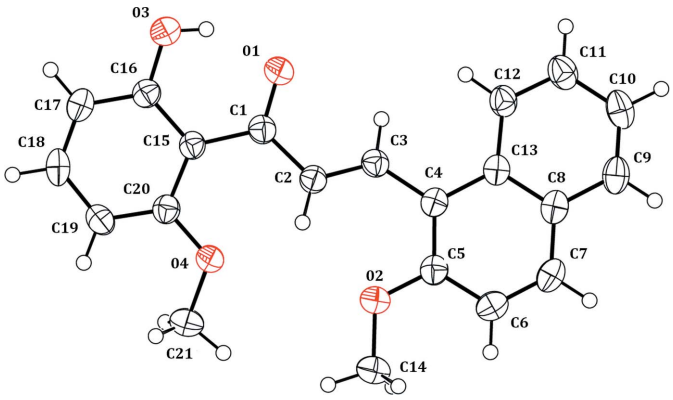


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

twisted [C21—O4—C20—C19 = 6.2 (3)°]. The dihedral angle formed between the plane of the naphthalene ring system (C4–C13; r.m.s. deviation = 0.014 Å) and the plane of the benzene ring (C15–C20; r.m.s. deviation = 0.006 Å) is 9.68 (1)°. An intramolecular O—H···O hydrogen bonds generates an *S*(6) ring motif. (Table 1).

Synthesis and crystallization

2-Hydroxy-6-methoxyacetophenone (830 mg, 5 mmol) was added to a solution of 2-methoxynaphthaldehyde (930 mg, 5 mmol) in 80 ml of ethanol (Fig. 2) and the temperature was adjusted to around 275–276 K in an ice-bath. To the cooled reaction mixture, 7 ml of 50% aqueous KOH solution were added, and the reaction mixture was stirred at room temperature for 20 h. This mixture was poured into iced water (150 ml) and was acidified (pH = 3) with 6 *N* HCl solution to give a precipitate. Filtration and washing with water afforded the crude solid of the title compound (802 mg, 48%). Recrystallization of the solid from ethanol solution gave orange blocks suitable for X-ray diffraction analysis (m.p. 472–473 K).

Refinement

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

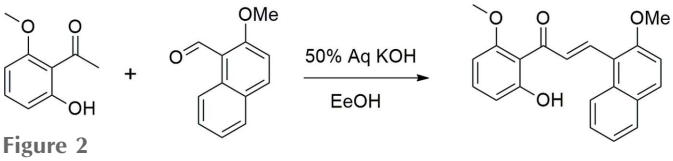


Figure 2
Synthetic scheme for the preparation of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O1	0.84	1.72	2.473 (2)	147

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{18}O_4$
M_r	334.35
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	200
a, *b*, *c* (Å)	14.8220 (9), 5.2416 (3), 20.8364 (13)
V (Å^3)	1618.80 (17)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.10
Crystal size (mm)	0.25 × 0.15 × 0.07
Data collection	
Diffractometer	Bruker CCD area detector
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11076, 2853, 2041
R_{int}	0.050
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.037, 0.088, 1.04
No. of reflections	2853
No. of parameters	229
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.20

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

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x191281 [https://doi.org/10.1107/S2414314619012811]

(*E*)-1-(2-Hydroxy-6-methoxyphenyl)-3-(2-methoxynaphthalen-1-yl)prop-2-en-1-one

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(*E*)-1-(2-Hydroxy-6-methoxyphenyl)-3-(2-methoxynaphthalen-1-yl)prop-2-en-1-one

Crystal data

$C_{21}H_{18}O_4$
M_r = 334.35
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 14.8220 (9) Å
b = 5.2416 (3) Å
c = 20.8364 (13) Å
V = 1618.80 (17) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4201 reflections
θ = 2.8–27.4°
μ = 0.10 mm^{-1}
T = 200 K
Block, orange
0.25 × 0.15 × 0.07 mm

Data collection

Bruker CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
11076 measured reflections
2853 independent reflections
2041 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.3°, θ_{min} = 2.0°
h = −19→19
k = −6→6
l = −27→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.088
S = 1.03
2853 reflections
229 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.60597 (14)	0.4619 (4)	0.12953 (12)	0.0341 (5)
O1	0.67919 (11)	0.3449 (3)	0.13586 (9)	0.0469 (5)
C2	0.59032 (15)	0.6894 (4)	0.16954 (12)	0.0336 (5)
H2	0.5448	0.8089	0.1583	0.040*
C3	0.64120 (15)	0.7258 (4)	0.22208 (12)	0.0330 (5)
H3	0.6855	0.5981	0.2293	0.040*
C4	0.63973 (15)	0.9290 (4)	0.27004 (12)	0.0328 (5)
C5	0.56895 (16)	1.1025 (4)	0.27589 (12)	0.0350 (5)
C6	0.56880 (17)	1.2901 (5)	0.32383 (13)	0.0407 (6)
H6	0.5193	1.4046	0.3270	0.049*
C7	0.63881 (17)	1.3101 (5)	0.36588 (12)	0.0405 (6)
H7	0.6379	1.4399	0.3977	0.049*
C8	0.71243 (16)	1.1411 (4)	0.36284 (11)	0.0360 (6)
C9	0.78445 (17)	1.1583 (5)	0.40781 (13)	0.0421 (6)
H9	0.7836	1.2895	0.4393	0.051*
C10	0.85437 (17)	0.9906 (5)	0.40658 (14)	0.0463 (7)
H10	0.9016	1.0026	0.4372	0.056*
C11	0.85583 (18)	0.7997 (5)	0.35945 (13)	0.0451 (7)
H11	0.9045	0.6817	0.3586	0.054*
C12	0.78887 (16)	0.7799 (5)	0.31489 (13)	0.0386 (6)
H12	0.7927	0.6515	0.2828	0.046*
C13	0.71324 (16)	0.9479 (4)	0.31534 (12)	0.0325 (5)
O2	0.49958 (11)	1.0813 (3)	0.23328 (9)	0.0441 (4)
C14	0.42411 (16)	1.2514 (4)	0.23981 (15)	0.0441 (6)
H14A	0.3956	1.2251	0.2817	0.066*
H14B	0.3802	1.2168	0.2057	0.066*
H14C	0.4451	1.4282	0.2364	0.066*
C15	0.53979 (15)	0.3670 (4)	0.08228 (11)	0.0313 (5)
C16	0.56510 (15)	0.1617 (4)	0.04161 (12)	0.0345 (5)
O3	0.64746 (11)	0.0559 (3)	0.04480 (9)	0.0463 (5)
H3A	0.6755	0.1164	0.0763	0.069*
C17	0.50611 (17)	0.0633 (5)	−0.00367 (13)	0.0418 (6)
H17	0.5247	−0.0727	−0.0308	0.050*
C18	0.42135 (19)	0.1621 (5)	−0.00917 (13)	0.0470 (7)
H18	0.3815	0.0945	−0.0406	0.056*
C19	0.39205 (16)	0.3592 (5)	0.03002 (13)	0.0406 (6)
H19	0.3325	0.4243	0.0257	0.049*
C20	0.44987 (16)	0.4601 (4)	0.07524 (12)	0.0344 (5)
O4	0.42414 (10)	0.6504 (3)	0.11552 (9)	0.0424 (4)
C21	0.33232 (16)	0.7297 (5)	0.11503 (16)	0.0550 (8)

H21A	0.3165	0.7922	0.0722	0.082*
H21B	0.3237	0.8666	0.1465	0.082*
H21C	0.2935	0.5849	0.1261	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0301 (12)	0.0399 (13)	0.0324 (14)	−0.0034 (10)	0.0024 (10)	−0.0007 (11)
O1	0.0320 (9)	0.0572 (11)	0.0516 (12)	0.0068 (8)	−0.0050 (8)	−0.0143 (10)
C2	0.0308 (12)	0.0363 (13)	0.0337 (14)	−0.0020 (9)	0.0016 (11)	−0.0022 (11)
C3	0.0292 (12)	0.0351 (12)	0.0348 (14)	−0.0029 (9)	0.0012 (11)	0.0003 (11)
C4	0.0342 (13)	0.0333 (12)	0.0310 (13)	−0.0041 (10)	0.0030 (11)	−0.0003 (11)
C5	0.0348 (13)	0.0386 (13)	0.0316 (13)	−0.0040 (10)	0.0030 (11)	−0.0002 (11)
C6	0.0437 (15)	0.0380 (14)	0.0404 (15)	0.0017 (11)	0.0066 (13)	−0.0008 (12)
C7	0.0515 (16)	0.0378 (14)	0.0323 (15)	−0.0056 (12)	0.0058 (13)	−0.0052 (11)
C8	0.0434 (14)	0.0366 (13)	0.0280 (14)	−0.0101 (10)	0.0040 (11)	0.0025 (11)
C9	0.0530 (16)	0.0431 (14)	0.0302 (13)	−0.0134 (12)	−0.0019 (13)	0.0012 (12)
C10	0.0502 (17)	0.0494 (16)	0.0392 (16)	−0.0134 (12)	−0.0126 (14)	0.0093 (14)
C11	0.0451 (16)	0.0381 (14)	0.0521 (19)	−0.0074 (11)	−0.0118 (13)	0.0055 (13)
C12	0.0405 (14)	0.0335 (12)	0.0416 (15)	−0.0054 (11)	−0.0066 (12)	0.0001 (12)
C13	0.0361 (13)	0.0321 (11)	0.0292 (12)	−0.0065 (10)	0.0012 (10)	0.0023 (11)
O2	0.0382 (9)	0.0487 (10)	0.0454 (11)	0.0100 (8)	−0.0047 (8)	−0.0078 (9)
C14	0.0355 (15)	0.0458 (14)	0.0511 (17)	0.0066 (11)	0.0009 (12)	0.0012 (14)
C15	0.0310 (12)	0.0362 (12)	0.0268 (13)	−0.0020 (10)	0.0017 (10)	−0.0007 (11)
C16	0.0361 (13)	0.0359 (12)	0.0315 (14)	0.0000 (10)	0.0022 (11)	0.0006 (11)
O3	0.0414 (10)	0.0532 (11)	0.0443 (12)	0.0087 (8)	−0.0011 (9)	−0.0117 (9)
C17	0.0493 (16)	0.0426 (14)	0.0335 (15)	−0.0026 (12)	−0.0002 (13)	−0.0071 (12)
C18	0.0523 (18)	0.0531 (15)	0.0355 (16)	−0.0089 (13)	−0.0113 (13)	−0.0088 (13)
C19	0.0336 (13)	0.0476 (14)	0.0406 (16)	0.0000 (11)	−0.0062 (12)	0.0008 (13)
C20	0.0352 (13)	0.0354 (12)	0.0326 (13)	−0.0021 (10)	0.0009 (11)	−0.0006 (11)
O4	0.0292 (8)	0.0507 (10)	0.0472 (11)	0.0045 (7)	−0.0037 (8)	−0.0137 (9)
C21	0.0328 (13)	0.0661 (18)	0.066 (2)	0.0101 (12)	−0.0014 (15)	−0.0152 (17)

Geometric parameters (Å, º)

C1—O1	1.254 (3)	C11—H11	0.9500
C1—O1	1.254 (3)	C12—C13	1.425 (3)
C1—C2	1.473 (3)	C12—H12	0.9500
C1—C15	1.476 (3)	O2—C14	1.437 (3)
C2—C3	1.343 (3)	C14—H14A	0.9800
C2—H2	0.9500	C14—H14B	0.9800
C3—C4	1.461 (3)	C14—H14C	0.9800
C3—H3	0.9500	C15—C16	1.420 (3)
C4—C5	1.394 (3)	C15—C20	1.427 (3)
C4—C13	1.445 (3)	C16—O3	1.343 (3)
C5—O2	1.363 (3)	C16—C17	1.386 (3)
C5—C6	1.402 (3)	O3—H3A	0.8400
C6—C7	1.362 (3)	C17—C18	1.364 (4)

C6—H6	0.9500	C17—H17	0.9500
C7—C8	1.407 (3)	C18—C19	1.386 (4)
C7—H7	0.9500	C18—H18	0.9500
C8—C13	1.416 (3)	C19—C20	1.379 (3)
C8—C9	1.423 (3)	C19—H19	0.9500
C9—C10	1.359 (3)	C20—O4	1.358 (3)
C9—H9	0.9500	O4—C21	1.423 (3)
C10—C11	1.402 (4)	C21—H21A	0.9800
C10—H10	0.9500	C21—H21B	0.9800
C11—C12	1.363 (3)	C21—H21C	0.9800

O1—C1—C2	118.2 (2)	C13—C12—H12	119.3
O1—C1—C2	118.2 (2)	C8—C13—C12	116.9 (2)
O1—C1—C15	118.7 (2)	C8—C13—C4	119.9 (2)
O1—C1—C15	118.7 (2)	C12—C13—C4	123.1 (2)
C2—C1—C15	123.1 (2)	C5—O2—C14	118.36 (19)
C3—C2—C1	119.2 (2)	O2—C14—H14A	109.5
C3—C2—H2	120.4	O2—C14—H14B	109.5
C1—C2—H2	120.4	H14A—C14—H14B	109.5
C2—C3—C4	130.8 (2)	O2—C14—H14C	109.5
C2—C3—H3	114.6	H14A—C14—H14C	109.5
C4—C3—H3	114.6	H14B—C14—H14C	109.5
C5—C4—C13	117.8 (2)	C16—C15—C20	116.4 (2)
C5—C4—C3	123.2 (2)	C16—C15—C1	118.6 (2)
C13—C4—C3	119.05 (19)	C20—C15—C1	125.0 (2)
O2—C5—C4	117.2 (2)	O3—C16—C17	117.0 (2)
O2—C5—C6	121.4 (2)	O3—C16—C15	121.6 (2)
C4—C5—C6	121.4 (2)	C17—C16—C15	121.4 (2)
C7—C6—C5	120.7 (2)	C16—O3—H3A	109.5
C7—C6—H6	119.6	C18—C17—C16	119.8 (2)
C5—C6—H6	119.6	C18—C17—H17	120.1
C6—C7—C8	120.9 (2)	C16—C17—H17	120.1
C6—C7—H7	119.5	C17—C18—C19	121.5 (2)
C8—C7—H7	119.5	C17—C18—H18	119.3
C7—C8—C13	119.2 (2)	C19—C18—H18	119.3
C7—C8—C9	120.8 (2)	C20—C19—C18	119.6 (2)
C13—C8—C9	119.9 (2)	C20—C19—H19	120.2
C10—C9—C8	121.2 (2)	C18—C19—H19	120.2
C10—C9—H9	119.4	O4—C20—C19	122.0 (2)
C8—C9—H9	119.4	O4—C20—C15	116.7 (2)
C9—C10—C11	119.1 (2)	C19—C20—C15	121.3 (2)
C9—C10—H10	120.4	C20—O4—C21	118.59 (19)
C11—C10—H10	120.4	O4—C21—H21A	109.5
C12—C11—C10	121.3 (2)	O4—C21—H21B	109.5
C12—C11—H11	119.3	H21A—C21—H21B	109.5
C10—C11—H11	119.3	O4—C21—H21C	109.5
C11—C12—C13	121.4 (2)	H21A—C21—H21C	109.5
C11—C12—H12	119.3	H21B—C21—H21C	109.5

C2—C1—O1—O1	0.00 (5)	C5—C4—C13—C8	−1.1 (3)
C15—C1—O1—O1	0.00 (6)	C3—C4—C13—C8	−179.0 (2)
O1—C1—C2—C3	−17.6 (3)	C5—C4—C13—C12	179.2 (2)
O1—C1—C2—C3	−17.6 (3)	C3—C4—C13—C12	1.3 (3)
C15—C1—C2—C3	162.7 (2)	C4—C5—O2—C14	177.6 (2)
C1—C2—C3—C4	−179.2 (2)	C6—C5—O2—C14	−2.0 (3)
C2—C3—C4—C5	13.8 (4)	O1—C1—C15—C16	−5.9 (3)
C2—C3—C4—C13	−168.4 (2)	O1—C1—C15—C16	−5.9 (3)
C13—C4—C5—O2	−179.4 (2)	C2—C1—C15—C16	173.7 (2)
C3—C4—C5—O2	−1.6 (3)	O1—C1—C15—C20	171.9 (2)
C13—C4—C5—C6	0.2 (3)	O1—C1—C15—C20	171.9 (2)
C3—C4—C5—C6	178.0 (2)	C2—C1—C15—C20	−8.4 (3)
O2—C5—C6—C7	−179.6 (2)	C20—C15—C16—O3	−178.8 (2)
C4—C5—C6—C7	0.8 (4)	C1—C15—C16—O3	−0.8 (3)
C5—C6—C7—C8	−0.9 (4)	C20—C15—C16—C17	1.8 (3)
C6—C7—C8—C13	−0.1 (3)	C1—C15—C16—C17	179.9 (2)
C6—C7—C8—C9	−178.4 (2)	O3—C16—C17—C18	179.8 (2)
C7—C8—C9—C10	177.8 (2)	C15—C16—C17—C18	−0.8 (4)
C13—C8—C9—C10	−0.6 (3)	C16—C17—C18—C19	−0.5 (4)
C8—C9—C10—C11	0.9 (4)	C17—C18—C19—C20	0.7 (4)
C9—C10—C11—C12	0.3 (4)	C18—C19—C20—O4	−178.9 (2)
C10—C11—C12—C13	−1.8 (4)	C18—C19—C20—C15	0.5 (4)
C7—C8—C13—C12	−179.2 (2)	C16—C15—C20—O4	177.75 (19)
C9—C8—C13—C12	−0.9 (3)	C1—C15—C20—O4	−0.1 (3)
C7—C8—C13—C4	1.1 (3)	C16—C15—C20—C19	−1.7 (3)
C9—C8—C13—C4	179.4 (2)	C1—C15—C20—C19	−179.5 (2)
C11—C12—C13—C8	2.0 (3)	C19—C20—O4—C21	6.2 (3)
C11—C12—C13—C4	−178.3 (2)	C15—C20—O4—C21	−173.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O1	0.84	1.72	2.473 (2)	147