

Crystal structure of (*E*)-3-(2,4-dimethoxyphenyl)-1-(1-hydroxynaphthalen-2-yl)prop-2-en-1-one

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In the title compound, $C_{21}H_{18}O_4$, the $C=C$ bond of the central enone group adopts an *E* conformation. The dihedral angle formed by the benzene ring and the naphthalene ring system is $6.60(2)^\circ$. The methoxy groups on the benzene ring are essentially coplanar with the ring; the $C-C-O-C$ torsion angles being $1.6(2)$ and $-177.1(1)^\circ$. The hydroxy group attached to the naphthalene ring is involved in an intramolecular $O-H\cdots O$ hydrogen bond. The relative conformation of the two double bonds in the enone group is *s-cisoid*. In the crystal, weak $C-H\cdots O$ hydrogen bonds link the molecules into chains propagating along $[010]$.

Keywords: crystal structure; chalcone; enone; benzochalcone; naphthalene.

CCDC reference: 1019819

1. Related literature

For the synthesis and biological properties of chalcone derivatives, see: Fuchigami *et al.* (2014); Kim *et al.* (2014); Mai *et al.* (2014); Smit & D'Na (2014). For details concerning benzo-chalcone derivatives, see: Juvale *et al.* (2013); Shin *et al.* (2013). For related structures, see: Ahn *et al.* (2013); Lim & Koh (2013).

2. Experimental

2.1. Crystal data

| | |
|-------------------------------|---|
| $C_{21}H_{18}O_4$ | $V = 3242.2(4) \text{ \AA}^3$ |
| $M_r = 334.35$ | $Z = 8$ |
| Monoclinic, $C2/c$ | Cu $K\alpha$ radiation |
| $a = 14.1148(9) \text{ \AA}$ | $\mu = 0.77 \text{ mm}^{-1}$ |
| $b = 14.7489(9) \text{ \AA}$ | $T = 147 \text{ K}$ |
| $c = 15.5929(10) \text{ \AA}$ | $0.19 \times 0.12 \times 0.08 \text{ mm}$ |
| $\beta = 92.804(4)^\circ$ | |

2.2. Data collection

| | |
|--|--|
| Bruker Kappa APEX DUO CCD diffractometer | 8800 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2007) | 2755 independent reflections |
| $T_{\min} = 0.669$, $T_{\max} = 0.753$ | 2505 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.026$ |

2.3. Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.095$ | |
| $S = 1.05$ | $\Delta\rho_{\max} = 0.15 \text{ e \AA}^{-3}$ |
| 2755 reflections | $\Delta\rho_{\min} = -0.27 \text{ e \AA}^{-3}$ |
| 232 parameters | |

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| O4—H4O \cdots O1 | 0.97 (2) | 1.59 (2) | 2.499 (1) | 155.1 (19) |
| C10—H10A \cdots O1 ⁱ | 0.98 | 2.57 | 3.538 (2) | 167 |
| C11—H11B \cdots O2 ⁱⁱ | 0.98 | 2.58 | 3.442 (2) | 147 |

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 2$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2770).

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supporting information

Acta Cryst. (2014). E70, o1034–o1035 [doi:10.1107/S1600536814018704]

Crystal structure of (*E*)-3-(2,4-dimethoxyphenyl)-1-(1-hydroxynaphthalen-2-yl)prop-2-en-1-one

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S1. Synthesis and crystallization

To a solution of 2,4-dimethoxybenzaldehyde (830 mg, 5 mmol) in 50 ml of ethanol was added 1-hydroxy-2-acetonaphthone (930 mg, 1 mmol) and the temperature was adjusted to around 275–276 K in an ice-bath. To the cooled reaction mixture 5 ml of 50% aqueous KOH solution was added, and the reaction mixture was stirred at room temperature for 24 h. This mixture was poured into iced water (100 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 (520 mg, 31%). Recrystallization of the solid from ethanol gave single crystals which were suitable for X-ray diffraction (M.p. 434–435 K).

S2. Refinement details

The OH H atom was located in a difference Fourier map and freely refined. The C-bound H atoms were placed in calculated positions and refined as riding atoms: C–H = 0.95 - 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $= 1.2U_{\text{eq}}(\text{C})$ for other H atoms.

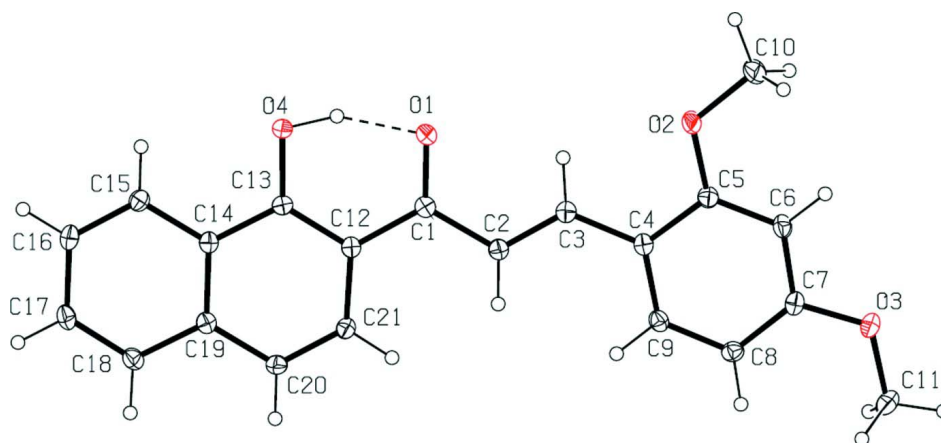
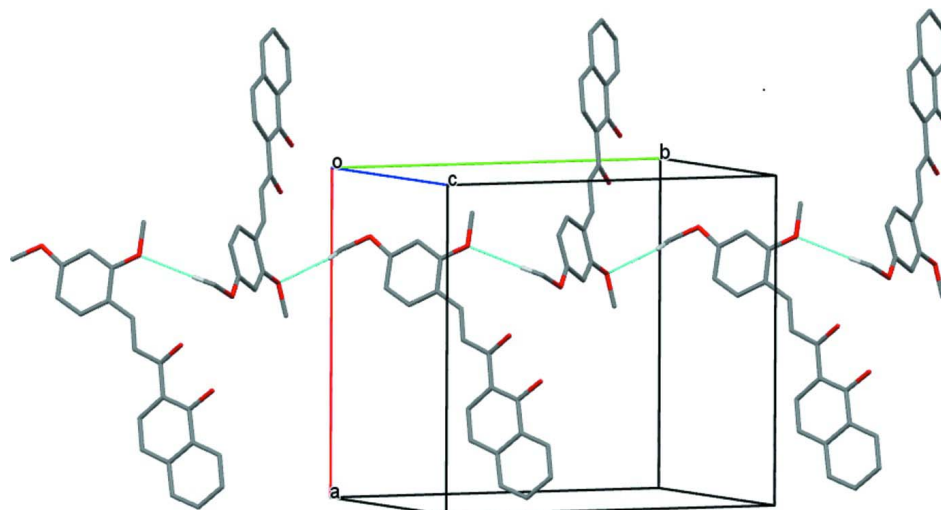


Figure 1

The molecular structure of the title molecule, with atom labelling. The displacement ellipsoids are drawn at the 50% probability level

**Figure 2**

A partial view along the *c* axis of the crystal packing of the title compound. The C—H···O hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity).

(*E*)-3-(2,4-Dimethoxyphenyl)-1-(1-hydroxynaphthalen-2-yl)prop-2-en-1-one

Crystal data

$C_{21}H_{18}O_4$

$M_r = 334.35$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 14.1148\ (9)\ \text{\AA}$

$b = 14.7489\ (9)\ \text{\AA}$

$c = 15.5929\ (10)\ \text{\AA}$

$\beta = 92.804\ (4)^\circ$

$V = 3242.2\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1408$

$D_x = 1.370\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 4341 reflections

$\theta = 4.3\text{--}66.4^\circ$

$\mu = 0.77\ \text{mm}^{-1}$

$T = 147\ \text{K}$

Needle, orange

$0.19 \times 0.12 \times 0.08\ \text{mm}$

Data collection

Bruker Kappa APEX DUO CCD
diffractometer

Radiation source: Bruker ImuS

Multi-layer optics monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2007)

$T_{\min} = 0.669$, $T_{\max} = 0.753$

8800 measured reflections

2755 independent reflections

2505 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 66.5^\circ$, $\theta_{\min} = 4.3^\circ$

$h = -16 \rightarrow 16$

$k = -17 \rightarrow 17$

$l = -13 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.095$

$S = 1.05$

2755 reflections

232 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 atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 1.958P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|--------------|----------------------------------|
| O1 | 0.46036 (6) | 0.17828 (6) | 1.03233 (6) | 0.0275 (2) |
| O2 | 0.19523 (6) | 0.11183 (7) | 0.85783 (7) | 0.0345 (3) |
| O3 | 0.17234 (6) | -0.10692 (6) | 0.63594 (6) | 0.0266 (2) |
| O4 | 0.59612 (6) | 0.24290 (6) | 1.12130 (6) | 0.0251 (2) |
| C1 | 0.51556 (9) | 0.13224 (8) | 0.98872 (8) | 0.0210 (3) |
| C2 | 0.47622 (9) | 0.07880 (8) | 0.91593 (8) | 0.0223 (3) |
| H2A | 0.5173 | 0.0432 | 0.8832 | 0.027* |
| C3 | 0.38286 (9) | 0.07983 (8) | 0.89525 (8) | 0.0213 (3) |
| H3A | 0.3454 | 0.1177 | 0.9294 | 0.026* |
| C4 | 0.33198 (8) | 0.03012 (8) | 0.82693 (8) | 0.0201 (3) |
| C5 | 0.23466 (9) | 0.04712 (8) | 0.80855 (8) | 0.0226 (3) |
| C6 | 0.18419 (9) | 0.00040 (9) | 0.74439 (8) | 0.0240 (3) |
| H6A | 0.1190 | 0.0134 | 0.7324 | 0.029* |
| C7 | 0.22926 (9) | -0.06572 (9) | 0.69744 (8) | 0.0215 (3) |
| C8 | 0.32474 (9) | -0.08523 (9) | 0.71418 (8) | 0.0233 (3) |
| H8A | 0.3557 | -0.1302 | 0.6822 | 0.028* |
| C9 | 0.37358 (9) | -0.03735 (9) | 0.77878 (8) | 0.0222 (3) |
| H9A | 0.4386 | -0.0512 | 0.7908 | 0.027* |
| C10 | 0.09670 (11) | 0.13031 (13) | 0.84355 (13) | 0.0516 (5) |
| H10A | 0.0772 | 0.1763 | 0.8845 | 0.077* |
| H10B | 0.0603 | 0.0746 | 0.8514 | 0.077* |
| H10C | 0.0847 | 0.1528 | 0.7849 | 0.077* |
| C11 | 0.21279 (10) | -0.17857 (10) | 0.58794 (9) | 0.0339 (3) |
| H11A | 0.1648 | -0.2029 | 0.5466 | 0.051* |
| H11B | 0.2349 | -0.2269 | 0.6272 | 0.051* |
| H11C | 0.2665 | -0.1551 | 0.5571 | 0.051* |
| C12 | 0.61749 (8) | 0.13277 (8) | 1.01100 (8) | 0.0193 (3) |
| C13 | 0.65294 (8) | 0.18928 (8) | 1.07709 (7) | 0.0190 (3) |
| C14 | 0.75210 (8) | 0.19224 (8) | 1.10073 (7) | 0.0193 (3) |
| C15 | 0.78867 (9) | 0.25063 (8) | 1.16617 (8) | 0.0220 (3) |
| H15A | 0.7470 | 0.2893 | 1.1952 | 0.026* |
| C16 | 0.88385 (9) | 0.25175 (9) | 1.18786 (8) | 0.0251 (3) |

| | | | | |
|------|-------------|-------------|-------------|------------|
| H16A | 0.9081 | 0.2916 | 1.2315 | 0.030* |
| C17 | 0.94584 (9) | 0.19415 (9) | 1.14567 (8) | 0.0259 (3) |
| H17A | 1.0117 | 0.1946 | 1.1616 | 0.031* |
| C18 | 0.91205 (9) | 0.13741 (9) | 1.08185 (8) | 0.0246 (3) |
| H18A | 0.9549 | 0.0991 | 1.0537 | 0.030* |
| C19 | 0.81422 (9) | 0.13491 (8) | 1.05720 (8) | 0.0203 (3) |
| C20 | 0.77706 (9) | 0.07710 (9) | 0.99071 (8) | 0.0228 (3) |
| H20A | 0.8185 | 0.0382 | 0.9617 | 0.027* |
| C21 | 0.68288 (9) | 0.07699 (8) | 0.96827 (8) | 0.0216 (3) |
| H21A | 0.6599 | 0.0387 | 0.9229 | 0.026* |
| H4O | 0.5337 (15) | 0.2284 (14) | 1.0965 (13) | 0.059 (6)* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|-------------|-------------|-------------|-------------|-------------|
| O1 | 0.0193 (4) | 0.0320 (5) | 0.0312 (5) | 0.0025 (4) | -0.0004 (4) | -0.0075 (4) |
| O2 | 0.0217 (5) | 0.0350 (6) | 0.0455 (6) | 0.0096 (4) | -0.0106 (4) | -0.0179 (5) |
| O3 | 0.0286 (5) | 0.0265 (5) | 0.0242 (5) | -0.0033 (4) | -0.0046 (4) | -0.0038 (4) |
| O4 | 0.0202 (5) | 0.0282 (5) | 0.0267 (5) | 0.0035 (4) | -0.0002 (4) | -0.0078 (4) |
| C1 | 0.0217 (6) | 0.0192 (6) | 0.0221 (6) | -0.0003 (5) | 0.0010 (5) | 0.0028 (5) |
| C2 | 0.0208 (6) | 0.0227 (6) | 0.0234 (6) | -0.0006 (5) | 0.0006 (5) | -0.0010 (5) |
| C3 | 0.0229 (6) | 0.0184 (6) | 0.0224 (6) | -0.0006 (5) | -0.0001 (5) | 0.0019 (5) |
| C4 | 0.0206 (6) | 0.0188 (6) | 0.0208 (6) | -0.0020 (5) | -0.0004 (5) | 0.0037 (5) |
| C5 | 0.0228 (6) | 0.0193 (6) | 0.0254 (6) | 0.0018 (5) | -0.0015 (5) | 0.0003 (5) |
| C6 | 0.0207 (6) | 0.0238 (7) | 0.0270 (7) | 0.0004 (5) | -0.0052 (5) | 0.0009 (5) |
| C7 | 0.0251 (6) | 0.0212 (6) | 0.0179 (6) | -0.0055 (5) | -0.0019 (5) | 0.0038 (5) |
| C8 | 0.0251 (6) | 0.0234 (7) | 0.0217 (6) | -0.0009 (5) | 0.0046 (5) | 0.0000 (5) |
| C9 | 0.0187 (6) | 0.0255 (7) | 0.0224 (6) | -0.0019 (5) | 0.0012 (5) | 0.0026 (5) |
| C10 | 0.0254 (8) | 0.0575 (11) | 0.0703 (12) | 0.0182 (7) | -0.0136 (7) | -0.0335 (9) |
| C11 | 0.0387 (8) | 0.0336 (8) | 0.0291 (7) | -0.0041 (6) | -0.0006 (6) | -0.0114 (6) |
| C12 | 0.0201 (6) | 0.0181 (6) | 0.0197 (6) | -0.0007 (5) | -0.0004 (5) | 0.0023 (5) |
| C13 | 0.0218 (6) | 0.0168 (6) | 0.0184 (6) | 0.0011 (5) | 0.0020 (5) | 0.0020 (5) |
| C14 | 0.0209 (6) | 0.0189 (6) | 0.0178 (6) | -0.0012 (5) | -0.0009 (5) | 0.0037 (5) |
| C15 | 0.0249 (6) | 0.0215 (6) | 0.0195 (6) | -0.0004 (5) | -0.0003 (5) | 0.0008 (5) |
| C16 | 0.0266 (7) | 0.0264 (7) | 0.0218 (6) | -0.0045 (5) | -0.0051 (5) | 0.0008 (5) |
| C17 | 0.0196 (6) | 0.0320 (7) | 0.0254 (6) | -0.0019 (5) | -0.0043 (5) | 0.0039 (5) |
| C18 | 0.0204 (6) | 0.0277 (7) | 0.0255 (7) | 0.0022 (5) | 0.0000 (5) | 0.0020 (5) |
| C19 | 0.0211 (6) | 0.0207 (6) | 0.0191 (6) | 0.0001 (5) | -0.0004 (5) | 0.0045 (5) |
| C20 | 0.0223 (6) | 0.0224 (6) | 0.0237 (6) | 0.0030 (5) | 0.0019 (5) | -0.0023 (5) |
| C21 | 0.0231 (6) | 0.0204 (6) | 0.0211 (6) | -0.0008 (5) | -0.0012 (5) | -0.0017 (5) |

Geometric parameters (Å, °)

| | | | |
|--------|-------------|----------|--------|
| O1—C1 | 1.2580 (15) | C10—H10B | 0.9800 |
| O2—C5 | 1.3612 (16) | C10—H10C | 0.9800 |
| O2—C10 | 1.4239 (17) | C11—H11A | 0.9800 |
| O3—C7 | 1.3635 (15) | C11—H11B | 0.9800 |
| O3—C11 | 1.4299 (17) | C11—H11C | 0.9800 |

| | | | |
|------------|-------------|---------------|-------------|
| O4—C13 | 1.3408 (15) | C12—C13 | 1.3987 (17) |
| O4—H4O | 0.97 (2) | C12—C21 | 1.4258 (17) |
| C1—C12 | 1.4634 (17) | C13—C14 | 1.4306 (17) |
| C1—C2 | 1.4683 (18) | C14—C15 | 1.4133 (17) |
| C2—C3 | 1.3414 (18) | C14—C19 | 1.4152 (17) |
| C2—H2A | 0.9500 | C15—C16 | 1.3693 (18) |
| C3—C4 | 1.4537 (17) | C15—H15A | 0.9500 |
| C3—H3A | 0.9500 | C16—C17 | 1.4059 (19) |
| C4—C9 | 1.3936 (18) | C16—H16A | 0.9500 |
| C4—C5 | 1.4119 (17) | C17—C18 | 1.3680 (19) |
| C5—C6 | 1.3832 (18) | C17—H17A | 0.9500 |
| C6—C7 | 1.3919 (18) | C18—C19 | 1.4156 (17) |
| C6—H6A | 0.9500 | C18—H18A | 0.9500 |
| C7—C8 | 1.3902 (18) | C19—C20 | 1.4227 (18) |
| C8—C9 | 1.3858 (18) | C20—C21 | 1.3580 (17) |
| C8—H8A | 0.9500 | C20—H20A | 0.9500 |
| C9—H9A | 0.9500 | C21—H21A | 0.9500 |
| C10—H10A | 0.9800 | | |
| | | | |
| C5—O2—C10 | 117.97 (11) | O3—C11—H11A | 109.5 |
| C7—O3—C11 | 117.44 (10) | O3—C11—H11B | 109.5 |
| C13—O4—H4O | 102.7 (12) | H11A—C11—H11B | 109.5 |
| O1—C1—C12 | 119.77 (11) | O3—C11—H11C | 109.5 |
| O1—C1—C2 | 119.13 (11) | H11A—C11—H11C | 109.5 |
| C12—C1—C2 | 121.11 (11) | H11B—C11—H11C | 109.5 |
| C3—C2—C1 | 120.65 (11) | C13—C12—C21 | 118.15 (11) |
| C3—C2—H2A | 119.7 | C13—C12—C1 | 119.35 (11) |
| C1—C2—H2A | 119.7 | C21—C12—C1 | 122.50 (11) |
| C2—C3—C4 | 127.94 (12) | O4—C13—C12 | 121.93 (11) |
| C2—C3—H3A | 116.0 | O4—C13—C14 | 116.85 (11) |
| C4—C3—H3A | 116.0 | C12—C13—C14 | 121.22 (11) |
| C9—C4—C5 | 116.67 (11) | C15—C14—C19 | 119.82 (11) |
| C9—C4—C3 | 123.29 (11) | C15—C14—C13 | 121.53 (11) |
| C5—C4—C3 | 120.00 (11) | C19—C14—C13 | 118.65 (11) |
| O2—C5—C6 | 123.18 (11) | C16—C15—C14 | 120.33 (12) |
| O2—C5—C4 | 115.50 (11) | C16—C15—H15A | 119.8 |
| C6—C5—C4 | 121.32 (12) | C14—C15—H15A | 119.8 |
| C5—C6—C7 | 119.80 (11) | C15—C16—C17 | 120.18 (12) |
| C5—C6—H6A | 120.1 | C15—C16—H16A | 119.9 |
| C7—C6—H6A | 120.1 | C17—C16—H16A | 119.9 |
| O3—C7—C8 | 124.91 (11) | C18—C17—C16 | 120.48 (12) |
| O3—C7—C6 | 114.42 (11) | C18—C17—H17A | 119.8 |
| C8—C7—C6 | 120.67 (11) | C16—C17—H17A | 119.8 |
| C9—C8—C7 | 118.31 (12) | C17—C18—C19 | 120.94 (12) |
| C9—C8—H8A | 120.8 | C17—C18—H18A | 119.5 |
| C7—C8—H8A | 120.8 | C19—C18—H18A | 119.5 |
| C8—C9—C4 | 123.21 (12) | C14—C19—C18 | 118.24 (11) |
| C8—C9—H9A | 118.4 | C14—C19—C20 | 119.51 (11) |

| | | | |
|---------------|--------------|-----------------|--------------|
| C4—C9—H9A | 118.4 | C18—C19—C20 | 122.24 (11) |
| O2—C10—H10A | 109.5 | C21—C20—C19 | 120.66 (11) |
| O2—C10—H10B | 109.5 | C21—C20—H20A | 119.7 |
| H10A—C10—H10B | 109.5 | C19—C20—H20A | 119.7 |
| O2—C10—H10C | 109.5 | C20—C21—C12 | 121.79 (11) |
| H10A—C10—H10C | 109.5 | C20—C21—H21A | 119.1 |
| H10B—C10—H10C | 109.5 | C12—C21—H21A | 119.1 |
| | | | |
| O1—C1—C2—C3 | 0.91 (18) | C2—C1—C12—C21 | -4.86 (18) |
| C12—C1—C2—C3 | -179.00 (11) | C21—C12—C13—O4 | -179.06 (10) |
| C1—C2—C3—C4 | -178.69 (11) | C1—C12—C13—O4 | 0.41 (17) |
| C2—C3—C4—C9 | 9.1 (2) | C21—C12—C13—C14 | 0.81 (17) |
| C2—C3—C4—C5 | -173.07 (12) | C1—C12—C13—C14 | -179.73 (10) |
| C10—O2—C5—C6 | 1.6 (2) | O4—C13—C14—C15 | -1.54 (17) |
| C10—O2—C5—C4 | -178.52 (14) | C12—C13—C14—C15 | 178.59 (11) |
| C9—C4—C5—O2 | 178.52 (11) | O4—C13—C14—C19 | 178.34 (10) |
| C3—C4—C5—O2 | 0.59 (17) | C12—C13—C14—C19 | -1.53 (17) |
| C9—C4—C5—C6 | -1.56 (18) | C19—C14—C15—C16 | -0.24 (18) |
| C3—C4—C5—C6 | -179.48 (11) | C13—C14—C15—C16 | 179.64 (11) |
| O2—C5—C6—C7 | -179.17 (12) | C14—C15—C16—C17 | -0.57 (19) |
| C4—C5—C6—C7 | 0.91 (19) | C15—C16—C17—C18 | 0.90 (19) |
| C11—O3—C7—C8 | 3.18 (17) | C16—C17—C18—C19 | -0.38 (19) |
| C11—O3—C7—C6 | -177.12 (11) | C15—C14—C19—C18 | 0.74 (17) |
| C5—C6—C7—O3 | -179.88 (11) | C13—C14—C19—C18 | -179.15 (11) |
| C5—C6—C7—C8 | -0.17 (19) | C15—C14—C19—C20 | -179.25 (11) |
| O3—C7—C8—C9 | 179.82 (11) | C13—C14—C19—C20 | 0.86 (17) |
| C6—C7—C8—C9 | 0.13 (18) | C17—C18—C19—C14 | -0.43 (18) |
| C7—C8—C9—C4 | -0.87 (19) | C17—C18—C19—C20 | 179.56 (12) |
| C5—C4—C9—C8 | 1.56 (18) | C14—C19—C20—C21 | 0.52 (18) |
| C3—C4—C9—C8 | 179.41 (11) | C18—C19—C20—C21 | -179.47 (12) |
| O1—C1—C12—C13 | -4.21 (17) | C19—C20—C21—C12 | -1.29 (19) |
| C2—C1—C12—C13 | 175.70 (11) | C13—C12—C21—C20 | 0.62 (18) |
| O1—C1—C12—C21 | 175.23 (11) | C1—C12—C21—C20 | -178.83 (12) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| O4—H4O \cdots O1 | 0.97 (2) | 1.59 (2) | 2.499 (1) | 155.1 (19) |
| C10—H10A \cdots O1 ⁱ | 0.98 | 2.57 | 3.538 (2) | 167 |
| C11—H11B \cdots O2 ⁱⁱ | 0.98 | 2.58 | 3.442 (2) | 147 |

Symmetry codes: (i) $-x+1/2, -y+1/2, -z+2$; (ii) $-x+1/2, y-1/2, -z+3/2$.