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# (*E*)-3-[4-(Hexyloxy)phenyl]-1-(4-hydroxyphenyl)prop-2-en-1-one

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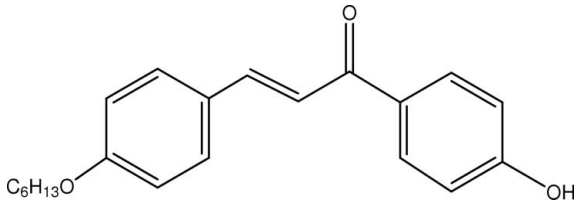
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.073; $wR$ factor = 0.139; data-to-parameter ratio = 25.5.

In the title compound, $C_{21}H_{24}O_3$, the enone group adopts an *s–cis* conformation. The planes of the aromatic rings are inclined at an angle of 6.1 (1)°. The alkoxy tail is not linear, with the maximum deviation from the least-squares plane being 0.375 (2) Å. Molecules are connected into extended chains along the *a* axis through O—H···$O_{carbonyl}$ hydrogen bonds and are interlinked *via* C—H···O interactions to form a two-dimensional array parallel to the *ab* plane.

## Related literature

For the biological properties of chalcone derivatives, see: Bhat *et al.* (2005); Xue *et al.* (2004); Zhao *et al.* (2005); Satyanarayana *et al.* (2004); Won *et al.* (2005). For related structures, see: Razak *et al.* (2009); Razak *et al.* (2009*a*,*b*); Ngaini, Fadzillah *et al.* (2009); Ngaini, Rahman *et al.* (2009). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).




## Experimental

### Crystal data

$C_{21}H_{24}O_3$
$M_r$ = 324.40
Orthorhombic, *Pbca*
$a$ = 10.0237 (2) Å
$b$ = 9.7695 (2) Å
$c$ = 35.3220 (6) Å
$V$ = 3458.96 (12) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 100 K
0.25 × 0.12 × 0.07 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.980, $T_{max}$ = 0.995
25370 measured reflections
5659 independent reflections
3311 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.086

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.139
$S$ = 1.02
5659 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1O1···O2$^{i}$ | 0.89 (3) | 1.77 (3) | 2.6466 (19) | 169 (2) |
| C1—H1$A$···O1$^{ii}$ | 0.93 | 2.55 | 3.458 (2) | 164 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2457).

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# supplementary materials

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# (*E*)-3-[4-(Hexyloxy)phenyl]-1-(4-hydroxyphenyl)prop-2-en-1-one

**I. A. Razak, H.-K. Fun, Z. Ngaini, N. I. A. Rahman and H. Hussain**

## Comment

Chalcones derivatives are reported to demonstrate biological properties such as an anti-malarial (Xue *et al.*, 2004), anti-cancer (Bhat *et al.*, 2005), anti-inflammatory (Won *et al.*, 2005), anti-platelet (Zhao *et al.*, 2005) as well as anti-hyperglycemic (Satyanarayana *et al.*, 2004) activities. Synthetic and naturally occurring chalcones have been extensively studied and developed as pharmaceutically important molecules. As part of our studies, we have synthesized the title chalcone derivative, (I), and tested its anti-bacterial activity against *E. coli* ATCC 8739; the compound showed anti-microbial activity. In this paper, we report the crystal structure of (I).

The conformation of the enone (O2/C7–C9) moiety in (I) is *s–cis* with the O2—C7—C8—C9 torsion angle being 5.7 (3)°. The mean plane through the enone moiety makes dihedral angles of 15.9 (1)° and 10.9 (1)°, with the C1–C6 and C10–C15 aromatic rings, respectively. The two aromatic rings form a dihedral angle of 6.1 (1)°.

The short H1A···H8A (2.16 Å) contact resulted in the slight widening of the C1—C6—C7 (123.0 (2)°) and C6—C7—C8 (120.7 (2)°) angles whereas the widening of C8—C9—C10 and C9—C10—C15 angles to 129.0 (2)° and 123.7 (2)° respectively, resulted from the close interatomic contact of H8A···H15A (2.34 Å). Correspondingly, the opening of the O3—C13—C12 (124.9 (2)°) angle is the consequence of strain induced by short H12A···H16A (2.28 Å) and H12A···H16B (2.38 Å) contacts. Similar features can also be found in previously reported related structures (Razak *et al.*, 2009; Razak *et al.*, 2009*a*,*b*; Ngaini, Fadzillah *et al.*, 2009; Ngaini, Rahman *et al.*, 2009).

Even though the C16—O3—C13—C12 torsion angle is 0.8 (3)°, only part of the alkoxyl tail, O3/C16–C18, is co-planar with the attached aromatic ring [maximum deviation of the least-squares plane of O3/C16–C18 is -0.002 (2) Å]. The alkoxyl chain is twisted about the C19—C20 bond as shown by the C18—C19—C20—C21 torsion angle being -73.4 (2)°. The least-squares plane through the the alkoxyl chain, O3/C16–C21, [maximum deviation of 0.375 (2) Å at C16] makes a dihedral angle of 49.1 (2)° with the attached aromatic ring.

In the crystal structure, intermolecular O1—H1O1···O2 hydrogen bonds between the hydroxy and keto groups link the molecules into extended chains along the *a* axis, Table 1. The chains are interlinked *via* C1—H1A···O1 interactions into a 2-D array parallel to the *ab*-plane, Table 1 and Fig. 2.

## Experimental

A mixture of 4-hydroxyacetophenone (1.36 g, 10 mmol), 4-hexyloxybenzaldehyde (2.06 ml, 10 mmol) and KOH (2.02 g, 36 mmol) in methanol (30 ml) was heated at reflux for 24 h. The reaction was cooled to room temperature and acidified with cold diluted HCl (2 N). The resulting precipitate was filtered, washed and dried. After redissolving in a hexane–ethanol (7:1) solution, followed by few days of slow evaporation, crystals were collected.

### Refinement

The O-bound H atom was located in a difference Fourier map and refined freely; O—H = 0.89 (3) Å. All the C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ (methyl-H atoms) and $1.2U_{eq}$ (other H atoms). The rotating model group was applied for the methyl group.

### Figures

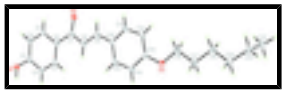

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme.

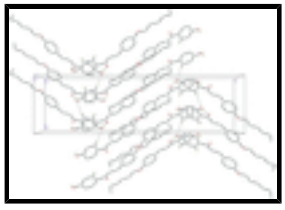

Fig. 2. The crystal packing in (I), viewed down the *a* axis. Intermolecular O—H···O hydrogen bonding and C—H···O contacts are shown as dashed lines. H atoms not involved in hydrogen bondings are omitted for clarity.

### (*E*)-3-[4-(Hexyloxy)phenyl]-1-(4-hydroxyphenyl)prop-2-en-1-one

*Crystal data*

| | |
|---|---|
| $C_{21}H_{24}O_3$ | $F_{000}$ = 1392 |
| $M_r$ = 324.40 | $D_x$ = 1.246 Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 2200 reflections |
| *a* = 10.0237 (2) Å | θ = 2.3–22.0º |
| *b* = 9.7695 (2) Å | μ = 0.08 mm$^{-1}$ |
| *c* = 35.3220 (6) Å | *T* = 100 K |
| *V* = 3458.96 (12) Å$^3$ | Block, colourless |
| *Z* = 8 | 0.25 × 0.12 × 0.07 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 5659 independent reflections |
| Radiation source: sealed tube | 3311 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.086 |
| *T* = 100 K | $\theta_{max}$ = 31.3º |
| φ and ω scans | $\theta_{min}$ = 1.2º |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | *h* = −14→14 |
| $T_{min}$ = 0.980, $T_{max}$ = 0.995 | *k* = −14→14 |
| 25370 measured reflections | *l* = −51→50 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.073$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.139$ | $w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 1.4637P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.02$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 5659 reflections | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| 222 parameters | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.45711 (14) | 0.93154 (14) | 0.31262 (3) | 0.0203 (3) |
| O2 | 0.17214 (13) | 0.81942 (14) | 0.15690 (3) | 0.0216 (3) |
| O3 | 0.47446 (13) | 0.24927 (13) | 0.00413 (4) | 0.0207 (3) |
| C1 | 0.43756 (18) | 0.74282 (19) | 0.22313 (5) | 0.0177 (4) |
| H1A | 0.4783 | 0.6696 | 0.2109 | 0.021* |
| C2 | 0.47890 (19) | 0.77884 (18) | 0.25929 (5) | 0.0183 (4) |
| H2A | 0.5452 | 0.7286 | 0.2714 | 0.022* |
| C3 | 0.42085 (18) | 0.89013 (19) | 0.27736 (5) | 0.0172 (4) |
| C4 | 0.32103 (19) | 0.96503 (19) | 0.25923 (5) | 0.0198 (4) |
| H4A | 0.2827 | 1.0402 | 0.2712 | 0.024* |
| C5 | 0.27945 (18) | 0.92715 (19) | 0.22348 (5) | 0.0193 (4) |
| H5A | 0.2127 | 0.9773 | 0.2116 | 0.023* |
| C6 | 0.33582 (18) | 0.81466 (18) | 0.20480 (5) | 0.0163 (4) |
| C7 | 0.28184 (18) | 0.77389 (19) | 0.16745 (5) | 0.0175 (4) |
| C8 | 0.35542 (18) | 0.67861 (19) | 0.14301 (5) | 0.0177 (4) |

| | | | | |
|---|---|---|---|---|
| H8A | 0.4336 | 0.6379 | 0.1516 | 0.021* |
| C9 | 0.30968 (18) | 0.65064 (19) | 0.10828 (5) | 0.0184 (4) |
| H9A | 0.2357 | 0.7010 | 0.1007 | 0.022* |
| C10 | 0.35965 (18) | 0.55225 (18) | 0.08071 (5) | 0.0169 (4) |
| C11 | 0.29114 (19) | 0.5369 (2) | 0.04661 (5) | 0.0201 (4) |
| H11A | 0.2197 | 0.5948 | 0.0415 | 0.024* |
| C12 | 0.32554 (19) | 0.43872 (19) | 0.02008 (5) | 0.0201 (4) |
| H12A | 0.2773 | 0.4303 | −0.0023 | 0.024* |
| C13 | 0.43319 (18) | 0.35286 (18) | 0.02730 (5) | 0.0176 (4) |
| C14 | 0.50604 (18) | 0.3682 (2) | 0.06095 (5) | 0.0193 (4) |
| H14A | 0.5795 | 0.3125 | 0.0655 | 0.023* |
| C15 | 0.46928 (18) | 0.46577 (19) | 0.08730 (5) | 0.0180 (4) |
| H15A | 0.5176 | 0.4743 | 0.1096 | 0.022* |
| C16 | 0.40164 (19) | 0.2288 (2) | −0.03062 (5) | 0.0207 (4) |
| H16A | 0.4010 | 0.3125 | −0.0454 | 0.025* |
| H16B | 0.3101 | 0.2034 | −0.0251 | 0.025* |
| C17 | 0.47013 (19) | 0.11601 (19) | −0.05233 (5) | 0.0211 (4) |
| H17A | 0.4708 | 0.0334 | −0.0371 | 0.025* |
| H17B | 0.5620 | 0.1420 | −0.0571 | 0.025* |
| C18 | 0.4013 (2) | 0.0863 (2) | −0.08992 (5) | 0.0229 (4) |
| H18A | 0.3874 | 0.1717 | −0.1033 | 0.027* |
| H18B | 0.3145 | 0.0462 | −0.0850 | 0.027* |
| C19 | 0.48161 (19) | −0.0107 (2) | −0.11501 (5) | 0.0219 (4) |
| H19A | 0.4997 | −0.0939 | −0.1009 | 0.026* |
| H19B | 0.5667 | 0.0318 | −0.1208 | 0.026* |
| C20 | 0.4129 (2) | −0.0490 (2) | −0.15221 (5) | 0.0274 (5) |
| H20A | 0.3798 | 0.0336 | −0.1642 | 0.033* |
| H20B | 0.4781 | −0.0899 | −0.1691 | 0.033* |
| C21 | 0.2977 (2) | −0.1482 (2) | −0.14688 (6) | 0.0286 (5) |
| H21A | 0.2614 | −0.1722 | −0.1711 | 0.043* |
| H21B | 0.2297 | −0.1057 | −0.1317 | 0.043* |
| H21C | 0.3291 | −0.2292 | −0.1344 | 0.043* |
| H1O1 | 0.529 (3) | 0.886 (3) | 0.3205 (7) | 0.057 (8)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0221 (7) | 0.0227 (7) | 0.0162 (6) | 0.0010 (6) | −0.0029 (6) | −0.0022 (6) |
| O2 | 0.0217 (7) | 0.0263 (7) | 0.0169 (6) | 0.0043 (6) | −0.0023 (5) | −0.0003 (6) |
| O3 | 0.0243 (7) | 0.0219 (7) | 0.0160 (6) | 0.0026 (6) | −0.0010 (5) | −0.0046 (5) |
| C1 | 0.0195 (9) | 0.0155 (9) | 0.0182 (9) | −0.0003 (8) | 0.0009 (8) | −0.0010 (7) |
| C2 | 0.0206 (10) | 0.0158 (9) | 0.0184 (9) | 0.0010 (8) | −0.0036 (8) | 0.0011 (7) |
| C3 | 0.0191 (9) | 0.0193 (9) | 0.0132 (8) | −0.0041 (7) | −0.0003 (7) | 0.0001 (7) |
| C4 | 0.0196 (9) | 0.0177 (9) | 0.0221 (9) | 0.0013 (8) | 0.0019 (8) | −0.0025 (8) |
| C5 | 0.0192 (9) | 0.0193 (9) | 0.0194 (9) | 0.0008 (8) | −0.0023 (7) | −0.0002 (8) |
| C6 | 0.0179 (9) | 0.0163 (8) | 0.0148 (8) | −0.0011 (7) | 0.0003 (7) | 0.0014 (7) |
| C7 | 0.0199 (9) | 0.0173 (9) | 0.0153 (9) | −0.0027 (8) | 0.0010 (7) | 0.0023 (7) |
| C8 | 0.0172 (9) | 0.0183 (9) | 0.0175 (9) | 0.0012 (8) | 0.0004 (7) | 0.0023 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0179 (9) | 0.0194 (9) | 0.0178 (9) | −0.0005 (7) | 0.0020 (7) | 0.0020 (8) |
| C10 | 0.0189 (9) | 0.0177 (9) | 0.0141 (8) | −0.0026 (7) | 0.0012 (7) | 0.0015 (7) |
| C11 | 0.0201 (10) | 0.0217 (10) | 0.0184 (9) | 0.0016 (8) | −0.0006 (8) | 0.0025 (8) |
| C12 | 0.0228 (10) | 0.0239 (10) | 0.0135 (8) | −0.0005 (8) | −0.0026 (8) | −0.0007 (8) |
| C13 | 0.0211 (9) | 0.0161 (9) | 0.0155 (9) | −0.0023 (7) | 0.0032 (7) | 0.0018 (7) |
| C14 | 0.0167 (9) | 0.0218 (9) | 0.0195 (9) | 0.0001 (8) | 0.0001 (7) | 0.0021 (8) |
| C15 | 0.0198 (9) | 0.0214 (9) | 0.0128 (8) | −0.0032 (8) | 0.0001 (7) | 0.0017 (7) |
| C16 | 0.0235 (10) | 0.0228 (10) | 0.0157 (9) | −0.0004 (8) | −0.0013 (8) | 0.0010 (8) |
| C17 | 0.0238 (10) | 0.0208 (9) | 0.0188 (9) | 0.0022 (8) | 0.0017 (8) | −0.0009 (8) |
| C18 | 0.0270 (11) | 0.0223 (10) | 0.0194 (9) | 0.0038 (8) | 0.0010 (8) | −0.0017 (8) |
| C19 | 0.0253 (10) | 0.0203 (9) | 0.0201 (9) | −0.0005 (8) | 0.0050 (8) | −0.0014 (8) |
| C20 | 0.0399 (13) | 0.0245 (10) | 0.0178 (10) | −0.0012 (10) | 0.0031 (9) | −0.0018 (8) |
| C21 | 0.0344 (12) | 0.0286 (11) | 0.0228 (10) | 0.0021 (10) | −0.0038 (9) | −0.0021 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C3 | 1.359 (2) | C12—C13 | 1.390 (3) |
| O1—H1O1 | 0.89 (3) | C12—H12A | 0.9300 |
| O2—C7 | 1.243 (2) | C13—C14 | 1.403 (2) |
| O3—C13 | 1.366 (2) | C14—C15 | 1.382 (3) |
| O3—C16 | 1.442 (2) | C14—H14A | 0.9300 |
| C1—C2 | 1.388 (2) | C15—H15A | 0.9300 |
| C1—C6 | 1.397 (2) | C16—C17 | 1.508 (3) |
| C1—H1A | 0.9300 | C16—H16A | 0.9700 |
| C2—C3 | 1.389 (2) | C16—H16B | 0.9700 |
| C2—H2A | 0.9300 | C17—C18 | 1.524 (3) |
| C3—C4 | 1.395 (3) | C17—H17A | 0.9700 |
| C4—C5 | 1.380 (2) | C17—H17B | 0.9700 |
| C4—H4A | 0.9300 | C18—C19 | 1.526 (3) |
| C5—C6 | 1.401 (2) | C18—H18A | 0.9700 |
| C5—H5A | 0.9300 | C18—H18B | 0.9700 |
| C6—C7 | 1.481 (2) | C19—C20 | 1.530 (3) |
| C7—C8 | 1.468 (3) | C19—H19A | 0.9700 |
| C8—C9 | 1.338 (2) | C19—H19B | 0.9700 |
| C8—H8A | 0.9300 | C20—C21 | 1.519 (3) |
| C9—C10 | 1.457 (2) | C20—H20A | 0.9700 |
| C9—H9A | 0.9300 | C20—H20B | 0.9700 |
| C10—C11 | 1.394 (2) | C21—H21A | 0.9600 |
| C10—C15 | 1.405 (3) | C21—H21B | 0.9600 |
| C11—C12 | 1.385 (3) | C21—H21C | 0.9600 |
| C11—H11A | 0.9300 | | |
| C3—O1—H1O1 | 110.8 (17) | C15—C14—H14A | 119.8 |
| C13—O3—C16 | 117.35 (14) | C13—C14—H14A | 119.8 |
| C2—C1—C6 | 121.13 (17) | C14—C15—C10 | 120.75 (17) |
| C2—C1—H1A | 119.4 | C14—C15—H15A | 119.6 |
| C6—C1—H1A | 119.4 | C10—C15—H15A | 119.6 |
| C1—C2—C3 | 119.76 (17) | O3—C16—C17 | 107.68 (15) |
| C1—C2—H2A | 120.1 | O3—C16—H16A | 110.2 |
| C3—C2—H2A | 120.1 | C17—C16—H16A | 110.2 |

| | | | |
|---|---|---|---|
| O1—C3—C2 | 122.84 (16) | O3—C16—H16B | 110.2 |
| O1—C3—C4 | 117.15 (16) | C17—C16—H16B | 110.2 |
| C2—C3—C4 | 120.01 (16) | H16A—C16—H16B | 108.5 |
| C5—C4—C3 | 119.72 (17) | C16—C17—C18 | 112.12 (16) |
| C5—C4—H4A | 120.1 | C16—C17—H17A | 109.2 |
| C3—C4—H4A | 120.1 | C18—C17—H17A | 109.2 |
| C4—C5—C6 | 121.30 (17) | C16—C17—H17B | 109.2 |
| C4—C5—H5A | 119.4 | C18—C17—H17B | 109.2 |
| C6—C5—H5A | 119.4 | H17A—C17—H17B | 107.9 |
| C1—C6—C5 | 118.05 (16) | C17—C18—C19 | 112.69 (16) |
| C1—C6—C7 | 123.00 (16) | C17—C18—H18A | 109.1 |
| C5—C6—C7 | 118.90 (16) | C19—C18—H18A | 109.1 |
| O2—C7—C8 | 119.66 (16) | C17—C18—H18B | 109.1 |
| O2—C7—C6 | 119.61 (16) | C19—C18—H18B | 109.1 |
| C8—C7—C6 | 120.73 (16) | H18A—C18—H18B | 107.8 |
| C9—C8—C7 | 119.77 (17) | C18—C19—C20 | 114.40 (17) |
| C9—C8—H8A | 120.1 | C18—C19—H19A | 108.7 |
| C7—C8—H8A | 120.1 | C20—C19—H19A | 108.7 |
| C8—C9—C10 | 129.03 (18) | C18—C19—H19B | 108.7 |
| C8—C9—H9A | 115.5 | C20—C19—H19B | 108.7 |
| C10—C9—H9A | 115.5 | H19A—C19—H19B | 107.6 |
| C11—C10—C15 | 117.62 (17) | C21—C20—C19 | 113.07 (16) |
| C11—C10—C9 | 118.60 (17) | C21—C20—H20A | 109.0 |
| C15—C10—C9 | 123.68 (16) | C19—C20—H20A | 109.0 |
| C12—C11—C10 | 122.42 (18) | C21—C20—H20B | 109.0 |
| C12—C11—H11A | 118.8 | C19—C20—H20B | 109.0 |
| C10—C11—H11A | 118.8 | H20A—C20—H20B | 107.8 |
| C11—C12—C13 | 119.14 (17) | C20—C21—H21A | 109.5 |
| C11—C12—H12A | 120.4 | C20—C21—H21B | 109.5 |
| C13—C12—H12A | 120.4 | H21A—C21—H21B | 109.5 |
| O3—C13—C12 | 124.91 (16) | C20—C21—H21C | 109.5 |
| O3—C13—C14 | 115.42 (16) | H21A—C21—H21C | 109.5 |
| C12—C13—C14 | 119.66 (17) | H21B—C21—H21C | 109.5 |
| C15—C14—C13 | 120.37 (18) | | |
| C6—C1—C2—C3 | −1.6 (3) | C8—C9—C10—C15 | 0.9 (3) |
| C1—C2—C3—O1 | −179.56 (16) | C15—C10—C11—C12 | 1.6 (3) |
| C1—C2—C3—C4 | 0.2 (3) | C9—C10—C11—C12 | −174.97 (17) |
| O1—C3—C4—C5 | −179.55 (16) | C10—C11—C12—C13 | −0.8 (3) |
| C2—C3—C4—C5 | 0.7 (3) | C16—O3—C13—C12 | 0.8 (3) |
| C3—C4—C5—C6 | −0.2 (3) | C16—O3—C13—C14 | 179.42 (15) |
| C2—C1—C6—C5 | 2.0 (3) | C11—C12—C13—O3 | 177.71 (17) |
| C2—C1—C6—C7 | −175.46 (17) | C11—C12—C13—C14 | −0.9 (3) |
| C4—C5—C6—C1 | −1.1 (3) | O3—C13—C14—C15 | −177.06 (16) |
| C4—C5—C6—C7 | 176.46 (17) | C12—C13—C14—C15 | 1.7 (3) |
| C1—C6—C7—O2 | 162.42 (17) | C13—C14—C15—C10 | −0.8 (3) |
| C5—C6—C7—O2 | −15.0 (3) | C11—C10—C15—C14 | −0.8 (3) |
| C1—C6—C7—C8 | −16.6 (3) | C9—C10—C15—C14 | 175.58 (17) |
| C5—C6—C7—C8 | 165.93 (16) | C13—O3—C16—C17 | 176.52 (15) |
| O2—C7—C8—C9 | 5.7 (3) | O3—C16—C17—C18 | −179.69 (15) |

| | | | |
|---|---|---|---|
| C6—C7—C8—C9 | −175.26 (17) | C16—C17—C18—C19 | 170.71 (16) |
| C7—C8—C9—C10 | −174.51 (17) | C17—C18—C19—C20 | 177.08 (17) |
| C8—C9—C10—C11 | 177.22 (19) | C18—C19—C20—C21 | −73.4 (2) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1O1···O2$^{i}$ | 0.89 (3) | 1.77 (3) | 2.6466 (19) | 169 (2) |
| C1—H1A···O1$^{ii}$ | 0.93 | 2.55 | 3.458 (2) | 164 |

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

**Fig. 1**

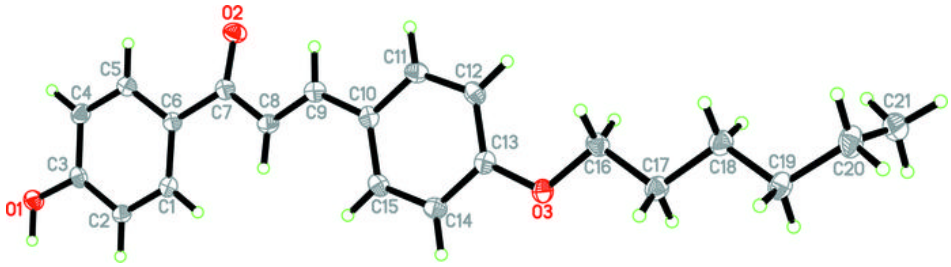
O2
C11
C12
C5
C4
C6
C7
C8
C9
C10
C13
C3
C16
C17
C18
C19
C20
C21
O1
C2
C1
C15
C14
O3

**Fig. 2**