

## (2-Hydroxy-7-methoxynaphthalen-1-yl)-(4-methylphenyl)methanone

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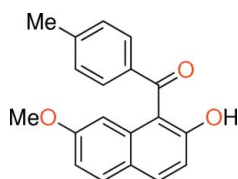
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Key indicators: single-crystal X-ray study;  $T = 193$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.106; data-to-parameter ratio = 13.4.

In the title compound,  $\text{C}_{19}\text{H}_{16}\text{O}_3$ , an intramolecular  $\text{O}-\text{H}\cdots\text{O}=\text{C}$  hydrogen bond is formed between the hydroxy and carbonyl groups on the naphthalene ring system, resulting in an  $S(6)$  ring. The angles between the  $\text{C}=\text{O}$  bond vector and the least-squares planes of the naphthalene ring system and the benzene ring are  $27.63$  (6) and  $47.99$  (7)°, respectively. The dihedral angle between the latter planes is  $61.39$  (5)°. In the crystal, two molecules are connected by pairs of intermolecular  $\text{O}-\text{H}\cdots\text{O}=\text{C}$  hydrogen bonds, forming centrosymmetric dimers with an  $R_2^2(4)$  graph-set motif. The molecular packing features  $\text{C}-\text{H}\cdots\pi$  interactions.

### Related literature

For electrophilic aromatic substitution of naphthalene derivatives, see: Okamoto & Yonezawa (2009). For the structures of closely related compounds, see: Mitsui *et al.* (2008); Nagasawa *et al.* (2010*a,b,c*). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



### Experimental

#### Crystal data

$\text{C}_{19}\text{H}_{16}\text{O}_3$   
 $M_r = 292.32$   
Monoclinic,  $P2_1/c$   
 $a = 11.1599$  (2) Å  
 $b = 6.05387$  (11) Å  
 $c = 22.0153$  (4) Å  
 $\beta = 90.317$  (1)°

$V = 1487.35$  (5) Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 0.71$  mm<sup>-1</sup>  
 $T = 193$  K  
 $0.60 \times 0.50 \times 0.30$  mm

#### Data collection

Rigaku R-Axis RAPID diffractometer  
Absorption correction: numerical (*NUMABS*; Higashi, 1999)  
 $T_{\min} = 0.676$ ,  $T_{\max} = 0.816$

25155 measured reflections  
2729 independent reflections  
2493 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.106$   
 $S = 1.04$   
2729 reflections  
204 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.15$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

CT1 and CT2 are the centroids of the C5–C10 and C12–C17 rings, respectively.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O3}$	0.95 (2)	1.70 (2)	2.5618 (14)	148 (2)
$\text{O1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.95 (2)	2.33 (2)	3.0083 (16)	128 (1)
$\text{C6}-\text{H6}\cdots\text{CT1}^{\text{ii}}$	0.95	2.71	3.5203 (13)	144
$\text{C17}-\text{H17}\cdots\text{CT1}^{\text{iii}}$	0.95	2.76	3.5492 (12)	141
$\text{C19}-\text{H19C}\cdots\text{CT2}^{\text{iv}}$	0.98	2.88	3.7834 (16)	154

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996), *ORTEP-3* for Windows (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2608).

### References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.  
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.  
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.  
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.  
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.  
Mitsui, R., Nakaema, K., Noguchi, K. & Yonezawa, N. (2008). *Acta Cryst.* **E64**, o2497.  
Nagasawa, A., Mitsui, R., Kato, Y., Okamoto, A. & Yonezawa, N. (2010*a*). *Acta Cryst.* **E66**, o2498.  
Nagasawa, A., Mitsui, R., Kato, Y., Okamoto, A. & Yonezawa, N. (2010*b*). *Acta Cryst.* **E66**, o2677.  
Nagasawa, A., Okamoto, A. & Yonezawa, N. (2010*c*). *Acta Cryst.* **E66**, o2738.  
Okamoto, A. & Yonezawa, N. (2009). *Chem. Lett.* **38**, 914–915.

Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.  
Rigaku/MSK (2004). *CrystalStructure*. Rigaku/MSK, The Woodlands, Texas,  
USA.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

**supplementary materials**

*Acta Cryst.* (2010). E66, o2820-o2821 [ doi:10.1107/S1600536810040614 ]

## (2-Hydroxy-7-methoxynaphthalen-1-yl)(4-methylphenyl)methanone

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### Comment

In the course of our study on selective *peri*-arylation of 2,7-dimethoxynaphthalene (Okamoto & Yonezawa, 2009), the crystal structures of several 1-monoarylated naphthalene compounds have been clarified along with 1,8-diaroynaphthalene. Furthermore, selective demethylation reaction of the 1-monoarylated 2,7-dimethoxynaphthalene compounds has been proved to yield the 1-monoaryl-2-hydroxynaphthalene compounds, which are rather susceptible to imination reaction. In this course, we recently reported crystal structure of several imine compounds prepared from 1-monoarylated naphthalene derivatives having 2-hydroxy group exemplified by 1-[(4-chlorophenyl)(phenylimino)methyl]-7-methoxy-2-naphthol-1,4-diazabicyclo[2.2.2]octane (2/1) (Nagasawa *et al.*, 2010a) and 1-[phenyl(3-nitrophenylimino)methyl]-7-methoxy-2-naphthol (Nagasawa *et al.*, 2010c) derived from (4-chlorophenyl)(2-hydroxy-7-methoxynaphthalen-1-yl)methanone (Mitsui *et al.*, 2008) and (2-hydroxy-7-methoxynaphthalen-1-yl)(phenyl)methanone (Nagasawa *et al.*, 2010b), respectively. As a part of our ongoing studies on the synthesis and crystal structure analysis of aryolated naphthalene homologues, we prepared and analysed the crystal structure of the title compound (I).

In the molecule (I), the intramolecular O—H $\cdots$ O=C hydrogen bond that forms a six-membered S(6) ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995) including carbonyl and hydroxy groups on the naphthalene ring (Table 1, Fig. 1). The conformation of these groups resembles to that of (2-hydroxy-7-methoxynaphthalen-1-yl)(phenyl)methanone (Nagasawa *et al.*, 2010b). The angles of C=O bond vector against the least-squares plane of the naphthalene ring (C1–C10) and benzene ring (C12–C17) are 27.63 (6) and 47.99 (7) $^\circ$ , respectively. The dihedral angle between the naphthalene ring (C1–C10) and benzene ring (C12–C17) is 61.39 (5) $^\circ$ .

In the crystal structure, O—H $\cdots$ O=C intermolecular hydrogen bonds between the hydroxy group and the carbonyl one on the naphthalene ring (Table 1) form a centrosymmetric dimer (Fig. 2) with a R<sub>2</sub><sup>2</sup>(4) graph set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995). The molecular packing of (I) is mainly stabilized by intermolecular C—H $\cdots$  $\pi$  hydrogen bond involving the centroid CT1 of the C5—C10 ring and the centroid CT2 of the C12–C17 phenyl ring. (Table 1).

### Experimental

To a solution of 1-(4-methylbenzoyl)-2,7-dimethoxynaphthalene (3.07 g, 10 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (100 ml) was added AlCl<sub>3</sub> (6.65 g, 50 mmol). The reaction mixture was refluxed for 30 min giving a dark red solution, which was then poured into H<sub>2</sub>O (30 ml). The aqueous layer was extracted with CHCl<sub>3</sub> (30 ml  $\times$  3). The combined organic layers were washed with brine (30 ml  $\times$  3) and dried over MgSO<sub>4</sub> overnight. The solvent was removed *in vacuo* and the crude material was purified by recrystallization from hexane to give compound (I) as yellow platelets (m.p. 385.5–386.00 K, yield 1.76 g, 60%).

Spectroscopic Data: <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  11.40 (s, 1H), 7.83 (d, *J* = 8.9 Hz, 1H), 7.61 (d, *J* = 8.6 Hz, 1H), 7.52 (d, *J* = 7.9 Hz, 2H), 7.22 (d, *J* = 7.9 Hz, 2H), 7.07 (d, *J* = 8.9 Hz, 1H), 6.89 (dd, *J* = 8.9, 2.4 Hz, 1H), 6.64 (d, *J* = 2.4 Hz, 1H), 3.31 (s, 3H), 2.41 (s, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  200.0, 162.3, 158.1, 143.2, 138.0, 136.1, 134.2, 130.0,

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129.5, 129.3, 123.8, 116.5, 115.8, 114.0, 106.6, 54.5, 21.7; IR (KBr): 3443, 2929, 1620, 1561, 1514, 1233; HRMS ( $m/z$ ):  $[M + H]^+$  calcd for  $C_{19}H_{17}O_3$ , 293.1178; found, 293.1189.

### Refinement

All the H-atoms could be located in difference Fourier maps. The coordinates of the OH hydrogen atom were refined using restraint ( $O1-H1 = 0.95 (2) \text{ \AA}$ ) with  $U_{iso}(H) = 1.5U_{eq}(O)$ . The H atoms attached to carbon were introduced in calculated positions and treated as riding on their parent atoms with  $C-H = 0.98 \text{ \AA}$  (methyl) or  $0.95 \text{ \AA}$  (aromatic) with  $U_{iso}(H) = 1.2U_{eq}(C_{aromatic})$  or  $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$ .

### Figures

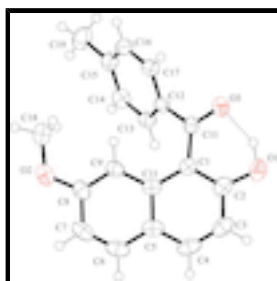


Fig. 1. The asymmetric unit of compound (I) with the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability. H atoms are represented as small spheres of arbitrary radii. Intramolecular hydrogen bond is shown as a dashed line.

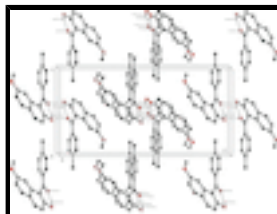


Fig. 2. A crystal packing diagram of compound (I), viewed down the  $b$  axis. Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity.

### (2-Hydroxy-7-methoxynaphthalen-1-yl)(4-methylphenyl)methanone

#### Crystal data

$C_{19}H_{16}O_3$

$M_r = 292.32$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P 2ybc$

$a = 11.1599 (2) \text{ \AA}$

$b = 6.05387 (11) \text{ \AA}$

$c = 22.0153 (4) \text{ \AA}$

$\beta = 90.317 (1)^\circ$

$V = 1487.35 (5) \text{ \AA}^3$

$Z = 4$

$F(000) = 616$

$D_x = 1.305 \text{ Mg m}^{-3}$

Melting point =  $385.5\text{--}386.0 \text{ K}$

Cu  $K\alpha$  radiation,  $\lambda = 1.54187 \text{ \AA}$

Cell parameters from 21159 reflections

$\theta = 4.0\text{--}68.2^\circ$

$\mu = 0.71 \text{ mm}^{-1}$

$T = 193 \text{ K}$

Block, colorless

$0.60 \times 0.50 \times 0.30 \text{ mm}$

#### Data collection

Rigaku R-Axis RAPID

2729 independent reflections

diffractometer	
Radiation source: rotating anode	2493 reflections with $I > 2\sigma(I)$
graphite	$R_{\text{int}} = 0.029$
Detector resolution: 10.00 pixels $\text{mm}^{-1}$	$\theta_{\text{max}} = 68.2^\circ$ , $\theta_{\text{min}} = 4.0^\circ$
$\omega$ scans	$h = -13 \rightarrow 13$
Absorption correction: numerical (NUMABS; Higashi, 1999)	$k = -7 \rightarrow 7$
$T_{\text{min}} = 0.676$ , $T_{\text{max}} = 0.816$	$l = -26 \rightarrow 26$
25155 measured reflections	

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.106$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.4274P]$
2729 reflections	where $P = (F_o^2 + 2F_c^2)/3$
204 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
1 restraint	$\Delta\rho_{\text{max}} = 0.28 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$

### 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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.39263 (8)	0.16739 (19)	0.46840 (5)	0.0479 (3)
H1	0.4401 (16)	0.298 (3)	0.4719 (9)	0.072*
O2	0.83634 (10)	-0.07553 (19)	0.23277 (5)	0.0539 (3)
O3	0.56764 (8)	0.43447 (17)	0.45225 (4)	0.0464 (3)
C1	0.55371 (10)	0.1035 (2)	0.39700 (5)	0.0324 (3)
C2	0.44354 (11)	0.0500 (2)	0.42366 (6)	0.0391 (3)
C3	0.38007 (12)	-0.1414 (3)	0.40634 (7)	0.0476 (4)
H3	0.3062	-0.1761	0.4254	0.057*

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C4	0.42384 (12)	-0.2755 (3)	0.36282 (7)	0.0473 (4)
H4	0.3814	-0.4065	0.3527	0.057*
C5	0.53176 (12)	-0.2259 (2)	0.33185 (6)	0.0390 (3)
C6	0.57478 (13)	-0.3633 (2)	0.28499 (6)	0.0456 (4)
H6	0.5324	-0.4947	0.2752	0.055*
C7	0.67500 (14)	-0.3123 (2)	0.25359 (6)	0.0473 (4)
H7	0.7032	-0.4082	0.2226	0.057*
C8	0.73726 (12)	-0.1148 (2)	0.26736 (6)	0.0401 (3)
C9	0.69806 (11)	0.0239 (2)	0.31228 (5)	0.0344 (3)
H9	0.7394	0.1584	0.3198	0.041*
C10	0.59639 (11)	-0.0312 (2)	0.34765 (5)	0.0325 (3)
C11	0.62050 (10)	0.2897 (2)	0.42351 (5)	0.0316 (3)
C12	0.75391 (10)	0.3095 (2)	0.42076 (5)	0.0297 (3)
C13	0.82996 (11)	0.1359 (2)	0.43596 (5)	0.0340 (3)
H13	0.7978	-0.0045	0.4462	0.041*
C14	0.95292 (11)	0.1683 (2)	0.43615 (6)	0.0385 (3)
H14	1.0042	0.0491	0.4468	0.046*
C15	1.00296 (11)	0.3713 (2)	0.42111 (6)	0.0386 (3)
C16	0.92554 (12)	0.5426 (2)	0.40584 (6)	0.0399 (3)
H16	0.9577	0.6822	0.3949	0.048*
C17	0.80264 (11)	0.5137 (2)	0.40630 (6)	0.0349 (3)
H17	0.7513	0.6341	0.3967	0.042*
C18	0.90578 (16)	0.1156 (3)	0.24644 (8)	0.0635 (5)
H18A	0.8577	0.2483	0.2389	0.095*
H18B	0.9770	0.1183	0.2206	0.095*
H18C	0.9304	0.1118	0.2892	0.095*
C19	1.13644 (12)	0.4059 (3)	0.42216 (7)	0.0555 (4)
H19A	1.1608	0.4843	0.3853	0.083*
H19B	1.1769	0.2623	0.4240	0.083*
H19C	1.1586	0.4935	0.4579	0.083*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0303 (5)	0.0623 (7)	0.0510 (6)	0.0003 (4)	0.0060 (4)	0.0009 (5)
O2	0.0604 (6)	0.0584 (7)	0.0431 (5)	0.0026 (5)	0.0108 (5)	-0.0121 (5)
O3	0.0406 (5)	0.0471 (6)	0.0514 (6)	0.0026 (4)	0.0110 (4)	-0.0114 (5)
C1	0.0291 (6)	0.0354 (7)	0.0327 (6)	0.0000 (5)	-0.0054 (5)	0.0051 (5)
C2	0.0296 (6)	0.0481 (8)	0.0396 (7)	-0.0002 (6)	-0.0050 (5)	0.0082 (6)
C3	0.0324 (7)	0.0561 (9)	0.0541 (8)	-0.0112 (6)	-0.0057 (6)	0.0105 (7)
C4	0.0416 (7)	0.0441 (8)	0.0560 (8)	-0.0145 (6)	-0.0166 (6)	0.0096 (7)
C5	0.0435 (7)	0.0334 (7)	0.0398 (7)	-0.0028 (6)	-0.0157 (5)	0.0046 (5)
C6	0.0557 (9)	0.0342 (7)	0.0466 (8)	-0.0024 (6)	-0.0201 (6)	-0.0019 (6)
C7	0.0633 (9)	0.0395 (8)	0.0389 (7)	0.0102 (7)	-0.0136 (6)	-0.0106 (6)
C8	0.0470 (7)	0.0423 (8)	0.0309 (6)	0.0055 (6)	-0.0032 (5)	-0.0022 (5)
C9	0.0398 (7)	0.0318 (6)	0.0314 (6)	-0.0002 (5)	-0.0057 (5)	-0.0008 (5)
C10	0.0333 (6)	0.0318 (6)	0.0322 (6)	0.0004 (5)	-0.0096 (5)	0.0043 (5)
C11	0.0321 (6)	0.0348 (6)	0.0281 (6)	0.0017 (5)	0.0015 (4)	0.0013 (5)

C12	0.0318 (6)	0.0309 (6)	0.0263 (5)	-0.0006 (5)	-0.0018 (4)	-0.0041 (5)
C13	0.0373 (6)	0.0297 (6)	0.0350 (6)	-0.0010 (5)	-0.0019 (5)	0.0011 (5)
C14	0.0364 (7)	0.0419 (7)	0.0371 (6)	0.0080 (6)	-0.0061 (5)	0.0001 (5)
C15	0.0331 (6)	0.0498 (8)	0.0330 (6)	-0.0039 (6)	-0.0020 (5)	-0.0047 (6)
C16	0.0407 (7)	0.0354 (7)	0.0436 (7)	-0.0088 (6)	-0.0003 (5)	-0.0008 (6)
C17	0.0377 (7)	0.0293 (6)	0.0377 (6)	0.0008 (5)	-0.0033 (5)	-0.0015 (5)
C18	0.0617 (10)	0.0691 (11)	0.0600 (10)	-0.0069 (9)	0.0244 (8)	-0.0082 (8)
C19	0.0334 (7)	0.0789 (12)	0.0542 (9)	-0.0074 (7)	-0.0031 (6)	-0.0031 (8)

*Geometric parameters (Å, °)*

O1—C2	1.3433 (17)	C9—C10	1.4195 (18)
O1—H1	0.953 (15)	C9—H9	0.9500
O2—C8	1.3670 (17)	C11—C12	1.4953 (16)
O2—C18	1.424 (2)	C12—C17	1.3885 (18)
O3—C11	1.2332 (15)	C12—C13	1.3907 (17)
C1—C2	1.4031 (17)	C13—C14	1.3862 (18)
C1—C10	1.4414 (18)	C13—H13	0.9500
C1—C11	1.4704 (17)	C14—C15	1.390 (2)
C2—C3	1.410 (2)	C14—H14	0.9500
C3—C4	1.349 (2)	C15—C16	1.390 (2)
C3—H3	0.9500	C15—C19	1.5043 (18)
C4—C5	1.420 (2)	C16—C17	1.3827 (18)
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.411 (2)	C17—H17	0.9500
C5—C10	1.4237 (18)	C18—H18A	0.9800
C6—C7	1.354 (2)	C18—H18B	0.9800
C6—H6	0.9500	C18—H18C	0.9800
C7—C8	1.415 (2)	C19—H19A	0.9800
C7—H7	0.9500	C19—H19B	0.9800
C8—C9	1.3708 (18)	C19—H19C	0.9800
C2—O1—H1	105.1 (11)	O3—C11—C12	116.30 (11)
C8—O2—C18	117.68 (11)	C1—C11—C12	123.23 (11)
C2—C1—C10	118.60 (12)	C17—C12—C13	119.27 (11)
C2—C1—C11	117.02 (11)	C17—C12—C11	118.17 (11)
C10—C1—C11	124.33 (11)	C13—C12—C11	122.42 (11)
O1—C2—C1	124.04 (12)	C14—C13—C12	119.75 (12)
O1—C2—C3	114.79 (12)	C14—C13—H13	120.1
C1—C2—C3	121.11 (13)	C12—C13—H13	120.1
C4—C3—C2	120.21 (13)	C13—C14—C15	121.57 (12)
C4—C3—H3	119.9	C13—C14—H14	119.2
C2—C3—H3	119.9	C15—C14—H14	119.2
C3—C4—C5	121.68 (13)	C16—C15—C14	117.84 (12)
C3—C4—H4	119.2	C16—C15—C19	120.94 (13)
C5—C4—H4	119.2	C14—C15—C19	121.22 (13)
C6—C5—C4	121.28 (13)	C17—C16—C15	121.28 (12)
C6—C5—C10	119.50 (13)	C17—C16—H16	119.4
C4—C5—C10	119.21 (13)	C15—C16—H16	119.4
C7—C6—C5	121.63 (13)	C16—C17—C12	120.26 (12)



## supplementary materials

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C7—C6—H6	119.2	C16—C17—H17	119.9
C5—C6—H6	119.2	C12—C17—H17	119.9
C6—C7—C8	119.29 (13)	O2—C18—H18A	109.5
C6—C7—H7	120.4	O2—C18—H18B	109.5
C8—C7—H7	120.4	H18A—C18—H18B	109.5
O2—C8—C9	123.90 (13)	O2—C18—H18C	109.5
O2—C8—C7	115.18 (12)	H18A—C18—H18C	109.5
C9—C8—C7	120.92 (13)	H18B—C18—H18C	109.5
C8—C9—C10	120.74 (12)	C15—C19—H19A	109.5
C8—C9—H9	119.6	C15—C19—H19B	109.5
C10—C9—H9	119.6	H19A—C19—H19B	109.5
C9—C10—C5	117.76 (12)	C15—C19—H19C	109.5
C9—C10—C1	123.31 (11)	H19A—C19—H19C	109.5
C5—C10—C1	118.90 (12)	H19B—C19—H19C	109.5
O3—C11—C1	120.38 (11)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

CT1 and CT2 are the centroids of the C5–C10 and C12–C17 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O3	0.95 (2)	1.70 (2)	2.5618 (14)	148 (2)
O1—H1 $\cdots$ O3 <sup>i</sup>	0.95 (2)	2.33 (2)	3.0083 (16)	128 (1)
C6—H6 $\cdots$ CT1 <sup>ii</sup>	0.95	2.71	3.5203 (13)	144
C17—H17 $\cdots$ CT1 <sup>iii</sup>	0.95	2.76	3.5492 (12)	141
C19—H19C $\cdots$ CT2 <sup>iv</sup>	0.98	2.88	3.7834 (16)	154

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

Fig. 1

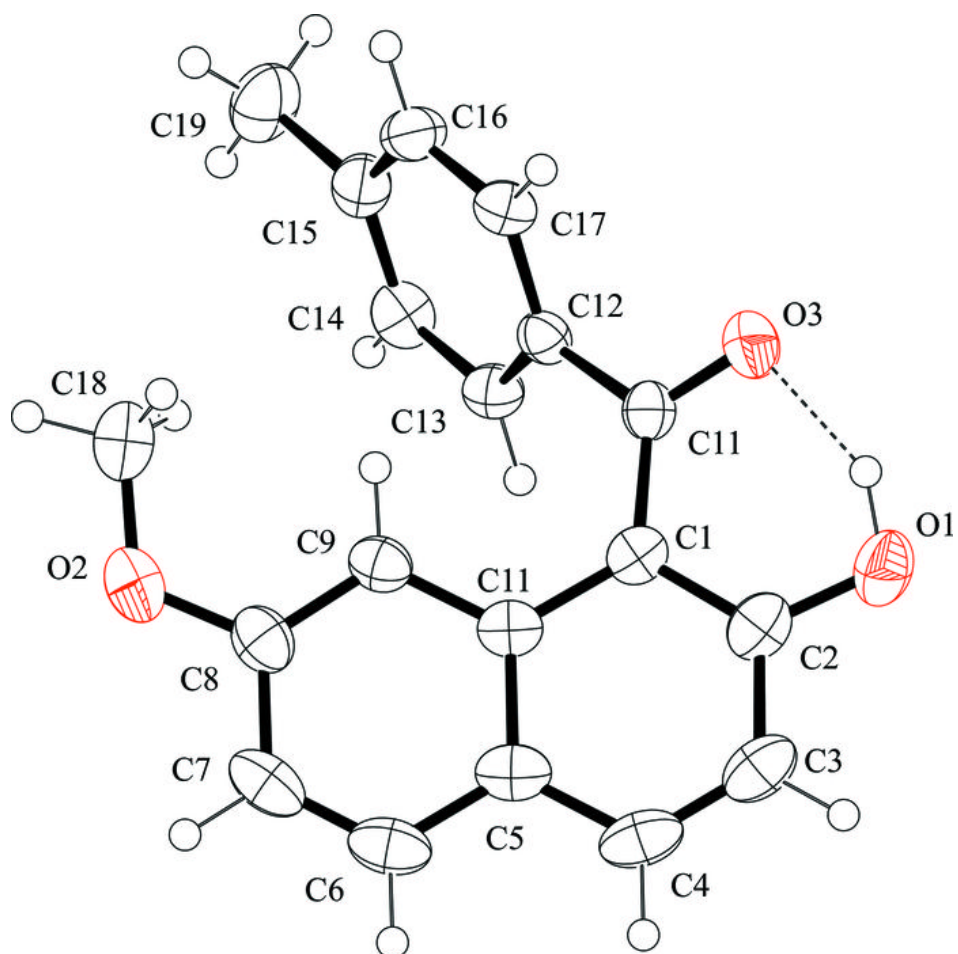


Fig. 2

