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# (3,6-Dimethoxynaphthalen-2-yl)(phenyl)methanone

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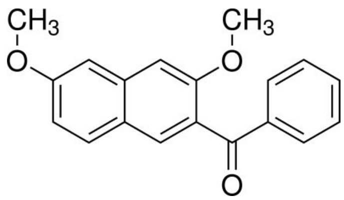
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.034; $wR$ factor = 0.098; data-to-parameter ratio = 13.5.

In the title compound, $C_{19}H_{16}O_3$, the dihedral angle between the naphthalene ring system and the phenyl ring is 68.32 (5)°. The bridging carbonyl C—C(=O)—C plane makes a dihedral angle of 54.32 (5)° with the naphthalene ring system and 21.45 (6)° with the phenyl ring. An intermolecular C—H⋯O hydrogen bond exists between the H atom of one methoxy group and the O atom of the second methoxy group in an adjacent molecule. The crystal packing is additionally stabilized by a weak C—H⋯O intermolecular interaction between an H atom of the naphthalene ring and the O atom of the carbonyl group.

## Related literature

For electrophilic aromatic substitution of naphthalene derivatives affording *peri*-aroylated compounds regioselectively, see: Okamoto & Yonezawa (2009). For the structures of closely related compounds, see: Kataoka *et al.* (2010); Kato *et al.* (2010); Muto *et al.* (2010); Nakaema, Okamoto *et al.* (2008); Nakaema, Watanabe *et al.* (2008); Nishijima *et al.* (2010); Watanabe *et al.* (2010).



## Experimental

*Crystal data*

$C_{19}H_{16}O_3$
$M_r$ = 292.32
Monoclinic, $P2_1/c$
$a$ = 8.7186 (2) Å
$b$ = 20.4650 (4) Å
$c$ = 8.5675 (2) Å
$\beta$ = 102.475 (1)°
$V$ = 1492.57 (6) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.71 mm$^{-1}$
$T$ = 193 K
0.60 × 0.50 × 0.20 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.677, $T_{max}$ = 0.872
26682 measured reflections
2735 independent reflections
2509 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
$S$ = 1.06
2735 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C4—H4⋯O1$^{i}$ | 0.95 | 2.58 | 3.4439 (13) | 151 |
| C18—H18$B$⋯O3$^{ii}$ | 0.98 | 2.42 | 3.3742 (15) | 164 |

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2010); program(s) used to solve structure: *Il Milione* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors would express their gratitude to Professor Keiichi Noguchi, Instrumentation Analysis Center, Tokyo University of Agriculture & Technology, for technical advice. This work was partially supported by the Mukai Science and Technology Foundation, Tokyo, Japan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FK2037).

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# supporting information

*Acta Cryst.* (2011). E**67**, o668 [doi:10.1107/S1600536811005630]

## (3,6-Dimethoxynaphthalen-2-yl)(phenyl)methanone

**Yuichi Kato, Ryo Takeuchi, Toyokazu Muto, Akiko Okamoto and Noriyuki Yonezawa**

### S1. Comment

In the course of our study on selective electrophilic aromatic aroylation of 2,7-dimethoxynaphthalene, *peri*-aroylnaphthalene compounds have proved to be formed regioselectively with the aid of suitable acidic mediator (Okamoto & Yonezawa, 2009). Recently, we have reported the structures of 1,8-diaroyl-2,7-dimethoxynaphthalenes such as 1,8-bis(4-methylbenzoyl)-2,7-dimethoxynaphthalene (Muto *et al.*, 2010) and 1,8-bis(4-aminobenzoyl)-2,7-dimethoxynaphthalene (Nishijima *et al.*, 2010). The aroyl groups at the 1,8-positions of the naphthalene rings in these compounds are bonded in a nearly perpendicular manner but the benzene rings of the aroyl groups tilt slightly toward the *exo* sides of the naphthalene rings. Such 1-aroylnaphthalene homologues as (2,7-dimethoxynaphthalen-1-yl)(3-nitrophenyl)methanone (Kataoka *et al.*, 2010) are also revealed to have essentially the same non-coplanar structure as observed for 1,8-diaroylated naphthalenes. Furthermore, we reported the crystal structure analysis of the corresponding *β*-isomers of 3-aroyl-2,7-dimethoxynaphthalenes such as 2-(4-chlorobenzoyl)-3,6-dimethoxynaphthalene (Nakaema, Okamoto *et al.*, 2008) and (4-fluorophenyl) (3,6-dimethoxy-2-naphthyl)methanone (Watanabe *et al.*, 2010). In the 3-aroylated naphthalenes, which are generally regarded to be thermodynamically more stable than the corresponding 1-positioned isomeric molecules, the aroyl groups are connected to the naphthalene rings in a moderately twisted fashion. On the other hand, there are several unique structural features in the benzoylated naphthalene homologues, 1-benzoyl-2,7-dimethoxynaphthalene (Kato *et al.*, 2010) and 1,8-dibenzoyl-2,7-dimethoxynaphthalene (Nakaema, Watanabe *et al.*, 2008). 1-Benzoyl-2,7-dimethoxynaphthalene contains three independent conformers and each of them forms a columnar structure, respectively. As a part of our ongoing study on the synthesis and structure of these homologous molecules, the crystal structure analysis of the title compound, a 3-monoaroylnaphthalene, is discussed in this article.

The molecular structure of the title molecule is displayed in Fig. 1. The benzene group is bonded to the naphthalene ring with a non-coplanar configuration. The dihedral angle between the best planes of the benzene ring (C12—C17) and the naphthalene ring (C1—C10) is 68.32 (5)°. The bridging carbonyl plane (O1—C3—C11—C12) makes a relatively large dihedral angle of 54.32 (5)° with the naphthalene ring (C1—C10) [C2—C3—C11—O1 torsion angle = -125.86 (12)°], whereas it makes a rather small dihedral angle of 21.45 (6)° with benzene ring (C12—C17) [O1—C11—C12—C13 torsion angle = -156.47 (11)°].

The crystal packing exhibits a weak C—H···O intermolecular interaction between the oxygen atom of the carbonyl group and the hydrogen atom of the naphthalene ring (Table 1, Fig. 2). The packing is additionally stabilized by a C—H···O hydrogen bond between the hydrogen of the 2-methoxy group, which is situated adjacent to the benzoyl group, and the ethereal oxygen atom of the 7-methoxy group in the neighboring molecule (Table 1, Fig. 3).

### S2. Experimental

A mixture of 2,7-dimethoxynaphthalene (3.74 g, 19.9 mmol), $FeCl_3$ (4.95 g, 37.1 mmol), trichloromethylbenzene (2.9 ml, 20 mmol) and dichloromethane (50 ml) was stirred at 293 K for 6 h, and the reaction mixture was poured into ice-cooled water followed by extraction with $CHCl_3$ (30 ml × 3). The combined extracts were washed with 2 *M* aqueous NaOH followed by washing with brine. The organic layer thus obtained was dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give cakes (yield 72%). The crude product was purified by flush silica gel chromatography ($CHCl_3$). Colorless platelet single crystals suitable for X-ray diffraction were obtained by crystallization from hexane and chloroform (yield 21%).

**Spectroscopic Data:**

$^1$H NMR $\delta$ (400 MHz, $CDCl_3$); 3.83 (3*H*, s), 3.95 (3*H*, s), 7.05 (1*H*, dd, *J* = 2.4, 9.2 Hz), 7.11 (1*H*, d, *J* = 2.4 Hz), 7.14 (1*H*, s), 7.44 (2*H*, t, *J* = 8.0 Hz), 7.56 (1*H*, t, *J* = 7.6 Hz), 7.69 (1*H*, d, *J* = 9.2 Hz), 7.78 (1*H*, s), 7.83–7.85 (2*H*, m) p.p.m..

$^{13}$C NMR $\delta$ (75 MHz, $CDCl_3$); 55.34, 55.54, 105.00, 105.38, 117.02, 123.15, 127.89, 128.18, 129.93, 130.01, 130.07, 132.87, 137.11, 138.06, 155.83, 159.30, 196.02 p.p.m..

IR (KBr): 1627 (C=O), 1580, 1502 (Ar, naphthalene), 1213 cm$^{-1}$.

HRMS (*m/z*): $[M + H]^+$ Calcd for $C_{19}H_{17}O_3$, 293.1178; found, 293.1203.

m.p. = 438.7–441.5 K.

## S3. Refinement

H atom positions were derived from geometrical considerations and were subsequently refined as riding atoms, with C—H = 0.95 (aromatic) and 0.98 (methyl) Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5(methyl).

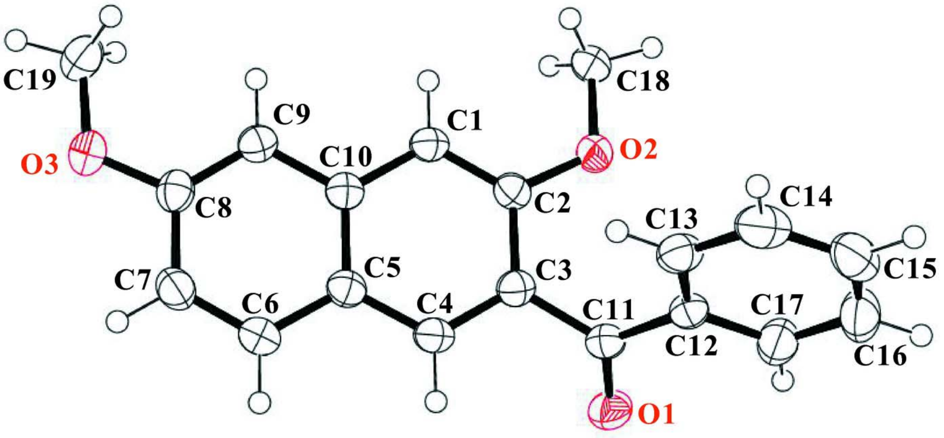

**Figure 1**

Molecular structure with displacement ellipsoids at 50% probability level for non-H atoms.

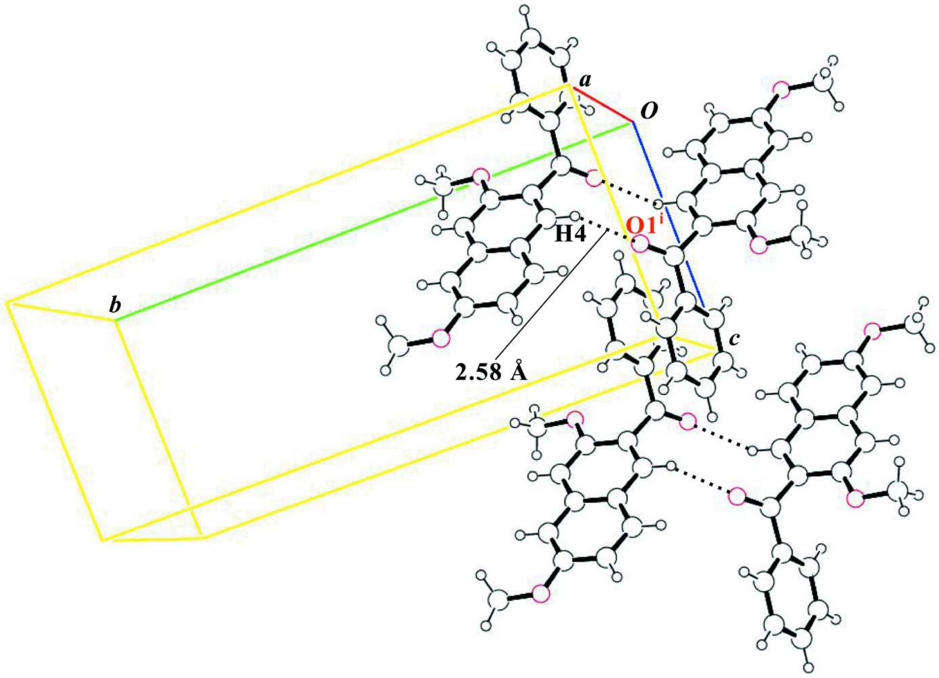

**Figure 2**

The C4—H4···O1 intermolecular interaction [symmetry code: (i) $-x+2, -y, -z+1$].

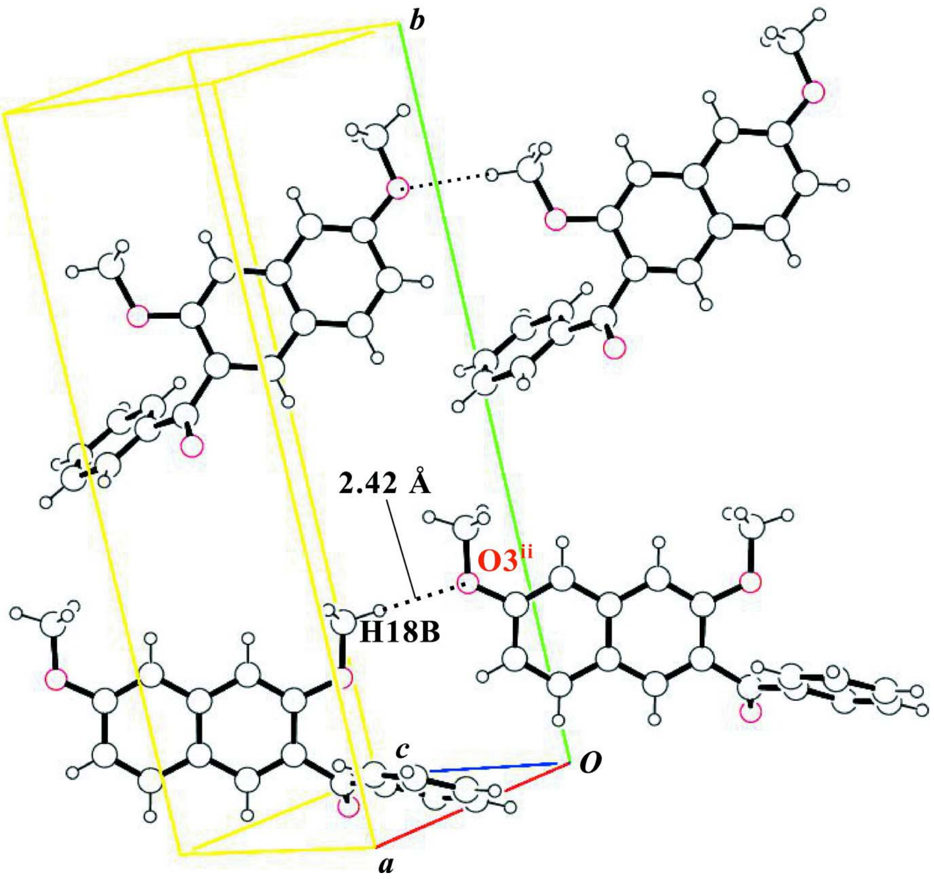

**Figure 3**

The C18—H18*B*···O3 intermolecular interaction [symmetry code: (ii) *x* – 1, *y*, *z* – 1].

## (3,6-Dimethoxynaphthalen-2-yl)(phenyl)methanone

### *Crystal data*

$C_{19}H_{16}O_3$
$M_r$ = 292.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 8.7186 (2) Å
$b$ = 20.4650 (4) Å
$c$ = 8.5675 (2) Å
$\beta$ = 102.475 (1)°
$V$ = 1492.57 (6) Å$^3$
$Z$ = 4

$F(000)$ = 616
$D_x$ = 1.301 Mg m$^{-3}$
Melting point = 438.7–441.5 K
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 16018 reflections
$\theta$ = 4.3–68.2°
$\mu$ = 0.71 mm$^{-1}$
$T$ = 193 K
Platelet, colorless
0.60 × 0.50 × 0.20 mm

### *Data collection*

Rigaku R-AXIS RAPID
diffractometer
Radiation source: rotating anode
Graphite monochromator
Detector resolution: 10.000 pixels mm$^{-1}$
$\omega$ scans

Absorption correction: numerical
(*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.677, $T_{max}$ = 0.872
26682 measured reflections
2735 independent reflections
2509 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044

| | |
|---|---|
| $\theta_{max} = 68.2°$, $\theta_{min} = 4.3°$ | $k = -24 \rightarrow 24$ |
| $h = -10 \rightarrow 10$ | $l = -10 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.034$ | $w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.2227P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.098$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.06$ | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |
| 2735 reflections | $\Delta\rho_{min} = -0.17$ e Å$^{-3}$ |
| 202 parameters | Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 0 restraints | Extinction coefficient: 0.0131 (8) |
| Primary atom site location: structure-invariant direct methods | |
| Secondary atom site location: difference Fourier map | |

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.78878 (10) | 0.03272 (4) | 0.31564 (10) | 0.0421 (2) |
| O2 | 0.82137 (9) | 0.20198 (4) | 0.16422 (9) | 0.0365 (2) |
| O3 | 1.49637 (9) | 0.28351 (4) | 0.80270 (10) | 0.0420 (2) |
| C1 | 1.04784 (12) | 0.23386 (5) | 0.36717 (13) | 0.0299 (2) |
| H1 | 1.0346 | 0.2786 | 0.3379 | 0.036* |
| C2 | 0.94725 (12) | 0.18810 (5) | 0.28459 (13) | 0.0297 (2) |
| C3 | 0.96544 (12) | 0.12068 (5) | 0.32677 (12) | 0.0301 (2) |
| C4 | 1.08301 (13) | 0.10241 (5) | 0.45329 (12) | 0.0314 (3) |
| H4 | 1.0931 | 0.0577 | 0.4834 | 0.038* |
| C5 | 1.18928 (12) | 0.14824 (5) | 0.53981 (12) | 0.0307 (3) |
| C6 | 1.31320 (13) | 0.13009 (5) | 0.66941 (13) | 0.0361 (3) |
| H6 | 1.3272 | 0.0854 | 0.6992 | 0.043* |
| C7 | 1.41212 (13) | 0.17582 (6) | 0.75144 (14) | 0.0378 (3) |
| H7 | 1.4948 | 0.1629 | 0.8374 | 0.045* |
| C8 | 1.39183 (12) | 0.24263 (6) | 0.70863 (13) | 0.0335 (3) |
| C9 | 1.27567 (12) | 0.26199 (5) | 0.58315 (13) | 0.0314 (3) |
| H9 | 1.2648 | 0.3069 | 0.5544 | 0.038* |
| C10 | 1.17134 (12) | 0.21514 (5) | 0.49575 (12) | 0.0288 (2) |
| C11 | 0.85594 (12) | 0.06982 (5) | 0.24066 (13) | 0.0312 (2) |
| C12 | 0.83398 (13) | 0.06328 (5) | 0.06375 (13) | 0.0320 (3) |
| C13 | 0.94512 (15) | 0.08664 (5) | −0.01647 (14) | 0.0386 (3) |

| | | | | |
|---|---|---|---|---|
| H13 | 1.0356 | 0.1089 | 0.0408 | 0.046* |
| C14 | 0.92417 (18) | 0.07753 (6) | −0.18018 (15) | 0.0495 (3) |
| H14 | 1.0017 | 0.0925 | −0.2343 | 0.059* |
| C15 | 0.7911 (2) | 0.04677 (6) | −0.26460 (16) | 0.0550 (4) |
| H15 | 0.7757 | 0.0418 | −0.3772 | 0.066* |
| C16 | 0.67992 (18) | 0.02314 (7) | −0.18521 (16) | 0.0546 (4) |
| H16 | 0.5884 | 0.0018 | −0.2432 | 0.066* |
| C17 | 0.70235 (15) | 0.03068 (6) | −0.02102 (15) | 0.0436 (3) |
| H17 | 0.6274 | 0.0135 | 0.0337 | 0.052* |
| C18 | 0.78092 (13) | 0.26937 (5) | 0.13734 (15) | 0.0371 (3) |
| H18A | 0.7644 | 0.2890 | 0.2367 | 0.044* |
| H18B | 0.6843 | 0.2730 | 0.0545 | 0.044* |
| H18C | 0.8663 | 0.2923 | 0.1024 | 0.044* |
| C19 | 1.47866 (16) | 0.35166 (6) | 0.77225 (16) | 0.0468 (3) |
| H19A | 1.4949 | 0.3611 | 0.6648 | 0.056* |
| H19B | 1.5563 | 0.3757 | 0.8514 | 0.056* |
| H19C | 1.3727 | 0.3652 | 0.7795 | 0.056* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0504 (5) | 0.0376 (4) | 0.0393 (5) | −0.0111 (4) | 0.0122 (4) | 0.0032 (3) |
| O2 | 0.0385 (4) | 0.0293 (4) | 0.0366 (4) | 0.0020 (3) | −0.0034 (3) | −0.0004 (3) |
| O3 | 0.0390 (5) | 0.0418 (5) | 0.0411 (5) | −0.0081 (3) | −0.0008 (4) | −0.0024 (4) |
| C1 | 0.0344 (5) | 0.0250 (5) | 0.0309 (6) | 0.0011 (4) | 0.0086 (4) | 0.0016 (4) |
| C2 | 0.0317 (5) | 0.0303 (5) | 0.0273 (5) | 0.0022 (4) | 0.0067 (4) | 0.0012 (4) |
| C3 | 0.0344 (5) | 0.0282 (5) | 0.0291 (5) | −0.0002 (4) | 0.0095 (4) | −0.0009 (4) |
| C4 | 0.0376 (6) | 0.0267 (5) | 0.0308 (6) | 0.0017 (4) | 0.0096 (5) | 0.0024 (4) |
| C5 | 0.0327 (5) | 0.0311 (5) | 0.0293 (5) | 0.0015 (4) | 0.0091 (4) | 0.0016 (4) |
| C6 | 0.0387 (6) | 0.0333 (6) | 0.0353 (6) | 0.0025 (4) | 0.0053 (5) | 0.0056 (4) |
| C7 | 0.0348 (6) | 0.0421 (6) | 0.0341 (6) | 0.0019 (5) | 0.0022 (5) | 0.0048 (5) |
| C8 | 0.0305 (5) | 0.0392 (6) | 0.0314 (6) | −0.0038 (4) | 0.0079 (4) | −0.0028 (4) |
| C9 | 0.0329 (5) | 0.0298 (5) | 0.0324 (6) | −0.0009 (4) | 0.0093 (4) | 0.0000 (4) |
| C10 | 0.0298 (5) | 0.0305 (5) | 0.0278 (5) | 0.0005 (4) | 0.0099 (4) | 0.0000 (4) |
| C11 | 0.0330 (5) | 0.0262 (5) | 0.0345 (6) | 0.0018 (4) | 0.0076 (4) | 0.0019 (4) |
| C12 | 0.0375 (6) | 0.0243 (5) | 0.0330 (6) | 0.0023 (4) | 0.0053 (4) | −0.0010 (4) |
| C13 | 0.0491 (7) | 0.0298 (5) | 0.0386 (6) | −0.0008 (5) | 0.0135 (5) | −0.0006 (5) |
| C14 | 0.0766 (9) | 0.0365 (6) | 0.0407 (7) | 0.0071 (6) | 0.0247 (7) | 0.0016 (5) |
| C15 | 0.0892 (11) | 0.0407 (7) | 0.0322 (7) | 0.0188 (7) | 0.0062 (7) | −0.0044 (5) |
| C16 | 0.0614 (8) | 0.0492 (8) | 0.0447 (8) | 0.0046 (6) | −0.0076 (6) | −0.0125 (6) |
| C17 | 0.0428 (6) | 0.0416 (6) | 0.0438 (7) | −0.0027 (5) | 0.0034 (5) | −0.0060 (5) |
| C18 | 0.0373 (6) | 0.0318 (6) | 0.0390 (6) | 0.0040 (4) | 0.0014 (5) | 0.0045 (4) |
| C19 | 0.0488 (7) | 0.0413 (7) | 0.0480 (7) | −0.0118 (5) | 0.0053 (6) | −0.0059 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C11 | 1.2224 (13) | C9—C10 | 1.4188 (15) |
| O2—C2 | 1.3637 (13) | C9—H9 | 0.9500 |

| | | | |
|---|---|---|---|
| O2—C18 | 1.4299 (13) | C11—C12 | 1.4921 (15) |
| O3—C8 | 1.3649 (13) | C12—C13 | 1.3884 (16) |
| O3—C19 | 1.4211 (15) | C12—C17 | 1.3891 (16) |
| C1—C2 | 1.3706 (15) | C13—C14 | 1.3873 (17) |
| C1—C10 | 1.4168 (15) | C13—H13 | 0.9500 |
| C1—H1 | 0.9500 | C14—C15 | 1.379 (2) |
| C2—C3 | 1.4265 (14) | C14—H14 | 0.9500 |
| C3—C4 | 1.3731 (15) | C15—C16 | 1.386 (2) |
| C3—C11 | 1.4948 (15) | C15—H15 | 0.9500 |
| C4—C5 | 1.4110 (15) | C16—C17 | 1.3864 (18) |
| C4—H4 | 0.9500 | C16—H16 | 0.9500 |
| C5—C10 | 1.4198 (14) | C17—H17 | 0.9500 |
| C5—C6 | 1.4212 (15) | C18—H18A | 0.9800 |
| C6—C7 | 1.3607 (17) | C18—H18B | 0.9800 |
| C6—H6 | 0.9500 | C18—H18C | 0.9800 |
| C7—C8 | 1.4166 (16) | C19—H19A | 0.9800 |
| C7—H7 | 0.9500 | C19—H19B | 0.9800 |
| C8—C9 | 1.3668 (16) | C19—H19C | 0.9800 |

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| C2—O2—C18 | 116.93 (8) | O1—C11—C3 | 120.03 (10) |
| C8—O3—C19 | 117.45 (9) | C12—C11—C3 | 119.46 (9) |
| C2—C1—C10 | 120.71 (9) | C13—C12—C17 | 119.52 (11) |
| C2—C1—H1 | 119.6 | C13—C12—C11 | 121.46 (10) |
| C10—C1—H1 | 119.6 | C17—C12—C11 | 118.97 (10) |
| O2—C2—C1 | 124.67 (9) | C14—C13—C12 | 120.04 (12) |
| O2—C2—C3 | 114.85 (9) | C14—C13—H13 | 120.0 |
| C1—C2—C3 | 120.42 (10) | C12—C13—H13 | 120.0 |
| C4—C3—C2 | 119.11 (10) | C15—C14—C13 | 120.24 (13) |
| C4—C3—C11 | 119.23 (9) | C15—C14—H14 | 119.9 |
| C2—C3—C11 | 121.62 (9) | C13—C14—H14 | 119.9 |
| C3—C4—C5 | 121.82 (10) | C14—C15—C16 | 120.00 (12) |
| C3—C4—H4 | 119.1 | C14—C15—H15 | 120.0 |
| C5—C4—H4 | 119.1 | C16—C15—H15 | 120.0 |
| C4—C5—C10 | 118.63 (10) | C15—C16—C17 | 119.92 (13) |
| C4—C5—C6 | 122.61 (10) | C15—C16—H16 | 120.0 |
| C10—C5—C6 | 118.76 (10) | C17—C16—H16 | 120.0 |
| C7—C6—C5 | 120.92 (10) | C16—C17—C12 | 120.23 (12) |
| C7—C6—H6 | 119.5 | C16—C17—H17 | 119.9 |
| C5—C6—H6 | 119.5 | C12—C17—H17 | 119.9 |
| C6—C7—C8 | 120.02 (10) | O2—C18—H18A | 109.5 |
| C6—C7—H7 | 120.0 | O2—C18—H18B | 109.5 |
| C8—C7—H7 | 120.0 | H18A—C18—H18B | 109.5 |
| O3—C8—C9 | 125.00 (10) | O2—C18—H18C | 109.5 |
| O3—C8—C7 | 114.12 (10) | H18A—C18—H18C | 109.5 |
| C9—C8—C7 | 120.88 (10) | H18B—C18—H18C | 109.5 |
| C8—C9—C10 | 120.06 (10) | O3—C19—H19A | 109.5 |
| C8—C9—H9 | 120.0 | O3—C19—H19B | 109.5 |
| C10—C9—H9 | 120.0 | H19A—C19—H19B | 109.5 |

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| C1—C10—C9 | 121.37 (10) | O3—C19—H19C | 109.5 |
| C1—C10—C5 | 119.28 (10) | H19A—C19—H19C | 109.5 |
| C9—C10—C5 | 119.34 (10) | H19B—C19—H19C | 109.5 |
| O1—C11—C12 | 120.47 (10) | | |

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| C18—O2—C2—C1 | −8.42 (15) | C8—C9—C10—C1 | 178.71 (10) |
| C18—O2—C2—C3 | 168.77 (9) | C8—C9—C10—C5 | −0.15 (15) |
| C10—C1—C2—O2 | 176.97 (9) | C4—C5—C10—C1 | 0.12 (15) |
| C10—C1—C2—C3 | −0.07 (16) | C6—C5—C10—C1 | −179.74 (9) |
| O2—C2—C3—C4 | −175.98 (9) | C4—C5—C10—C9 | 179.01 (9) |
| C1—C2—C3—C4 | 1.34 (16) | C6—C5—C10—C9 | −0.85 (15) |
| O2—C2—C3—C11 | 1.64 (14) | C4—C3—C11—O1 | 51.76 (14) |
| C1—C2—C3—C11 | 178.96 (9) | C2—C3—C11—O1 | −125.86 (11) |
| C2—C3—C4—C5 | −1.91 (16) | C4—C3—C11—C12 | −126.07 (10) |
| C11—C3—C4—C5 | −179.58 (9) | C2—C3—C11—C12 | 56.32 (14) |
| C3—C4—C5—C10 | 1.18 (16) | O1—C11—C12—C13 | −156.48 (11) |
| C3—C4—C5—C6 | −178.96 (10) | C3—C11—C12—C13 | 21.34 (15) |
| C4—C5—C6—C7 | −179.10 (10) | O1—C11—C12—C17 | 20.86 (15) |
| C10—C5—C6—C7 | 0.75 (16) | C3—C11—C12—C17 | −161.33 (10) |
| C5—C6—C7—C8 | 0.35 (17) | C17—C12—C13—C14 | 0.16 (17) |
| C19—O3—C8—C9 | 2.94 (16) | C11—C12—C13—C14 | 177.48 (10) |
| C19—O3—C8—C7 | −176.95 (10) | C12—C13—C14—C15 | 1.68 (18) |
| C6—C7—C8—O3 | 178.50 (10) | C13—C14—C15—C16 | −1.88 (19) |
| C6—C7—C8—C9 | −1.40 (17) | C14—C15—C16—C17 | 0.2 (2) |
| O3—C8—C9—C10 | −178.60 (9) | C15—C16—C17—C12 | 1.61 (19) |
| C7—C8—C9—C10 | 1.28 (16) | C13—C12—C17—C16 | −1.80 (17) |
| C2—C1—C10—C9 | −179.52 (9) | C11—C12—C17—C16 | −179.19 (11) |
| C2—C1—C10—C5 | −0.65 (15) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···O1$^{i}$ | 0.95 | 2.58 | 3.4439 (13) | 151 |
| C18—H18*B*···O3$^{ii}$ | 0.98 | 2.42 | 3.3742 (15) | 164 |

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