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# (8-Benzoyl-2,7-diethoxynaphthalen-1-yl)-(phenyl)methanone

**Atsumi Isogai, Takehiro Tsumuki, Shun Murohashi, Akiko Okamoto* and Noriyuki Yonezawa**

Department of Organic and Polymer Materials Chemistry, Tokyo University of Agriculture & Technology, 2-24-16 Naka-machi, Koganei, Tokyo 184-8588, Japan
Correspondence e-mail: aokamoto@cc.tuat.ac.jp

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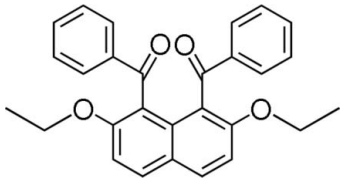
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.035; $wR$ factor = 0.091; data-to-parameter ratio = 14.0.

In the title compound, $C_{28}H_{24}O_4$, the benzoyl groups at the 1- and 8-positions of the naphthalene ring system are aligned almost antiparallel, and the benzene rings make a dihedral angle of 20.03 (7)°. The dihedral angles between the benzene rings and the naphthalene ring system are 68.42 (5) and 71.69 (5)°. In the crystal, adjacent molecules are linked *via* C—H···O hydrogen bonds, forming chains propagating along [100].

## Related literature

For electrophilic aroylation of naphthalene derivatives, see: Okamoto & Yonezawa (2009); Okamoto *et al.* (2011). For the structures of closely related compounds, see: Nakaema *et al.* (2008); Nishijima *et al.* (2010); Sasagawa *et al.* (2011); Tsumuki *et al.* (2011); Muto *et al.* (2012).



## Experimental

### Crystal data

$C_{28}H_{24}O_4$
$M_r$ = 424.47
Monoclinic, $P2_1/n$
$a$ = 7.92185 (14) Å
$b$ = 20.6794 (4) Å
$c$ = 14.2130 (3) Å
$\beta$ = 106.043 (1)°
$V$ = 2237.68 (7) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.67 mm$^{-1}$
$T$ = 193 K
0.60 × 0.50 × 0.10 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.689, $T_{max}$ = 0.936
39782 measured reflections
4076 independent reflections
3736 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
$S$ = 1.05
4076 reflections
292 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14···O3$^{i}$ | 0.95 | 2.37 | 3.2404 (16) | 153 |
| C21—H21···O4$^{ii}$ | 0.95 | 2.39 | 3.3326 (16) | 171 |

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2538).

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# supporting information

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## (8-Benzoyl-2,7-diethoxynaphthalen-1-yl)(phenyl)methanone

**Atsumi Isogai, Takehiro Tsumuki, Shun Murohashi, Akiko Okamoto and Noriyuki Yonezawa**

### S1. Comment

In the course of our study on selective electrophilic aromatic aroylation of naphthalene ring core, 1,8-diaroylnaphthalene compounds have proved to be formed regioselectively by the aid of a suitable acidic mediator (Okamoto & Yonezawa, 2009, Okamoto *et al.*, 2011). Recently, we have reported the X-ray crystal structure of 1,8-diaroyled 2,7-dimethoxynaphthalene derivatives such as 1,8-bis(4-aminobenzoyl)-2,7-dimethoxynaphthalene (Nishijima *et al.*, 2010), [8-(4-butoxybenzoyl)-2,7-dimethoxynaphthalen-1-yl](4-butoxyphenyl)methanone [1,8-bis(4-butoxybenzoyl)-2,7-dimethoxynaphthalene] (Sasagawa *et al.*, 2011), [2,7-dimethoxy-8-(2-naphthoyl)naphthalene-1-yl](naphthalene-2-yl)methanone [2,7-dimethoxy-1,8-bis(2-naphthoyl)naphthalene] (Tsumuki *et al.*, 2011), and (3,5-dimethylphenyl)[8-(3,5-dimethylbenzoyl)-2,7-dimethoxynaphthalen-1-yl]methanone (Muto *et al.*, 2012). The simplest molecule in these analogues, 1,8-dibenzoyl-2,7-dimethoxynaphthalene (Nakaema *et al.*, 2008), lies across a crystallographic 2-fold axis and the molecular packing is stabilized by C—H···O interactions between carbonyl groups and benzene ring and $\pi\cdots\pi$ interactions between benzene rings. As a part of our ongoing studies on the molecular structures of these kinds of homologous molecules, the X-ray crystal structure of the title compound, is reported on herein.

The molecule structure of the title molecule is illustrated in Fig. 1. The two benzoyl groups are situated in an (*anti*)-orientation and twisted away from the attached naphthalene ring. The dihedral angle between the planes of the two benzene rings (C12-C17 & C19-C24) is 20.03 (7)°. The dihedral angles between these benzene rings and the naphthalene (C1-C10) ring are 68.42 (5)° and 71.69 (5)°. These dihedral angles are similar to that reported for 1,8-dibenzoyl-2,7-dimethoxynaphthalene [80.25 (6)°; Nakaema *et al.*, 2008]. The torsion angles between the carbonyl groups and the naphthalene ring are -63.28 (14)° [C9—C1—C11—O3] and -66.19 (14)° [C9—C8—C18—O4]. The C═O groups lie almost in the plane of the attached benzene ring with torsion angles equal to 1.58 (17)° [O3—C11—C12—C17] and 1.44 (17)° [O4—C18—C19—C24].

In the crystal, the molecular packing of the title compound is mainly stabilized by two types of C—H···O interactions involving adjacent molecules and leading to the formation of chains along the *a* axis (Table 1 and Fig. 2).

### S2. Experimental

To a 100 ml flask, benzoyl chloride (422 mg, 3.0 mmol), titanium chloride (1.71 g, 9.0 mmol) and methylene chloride (1.5 ml) were stirred at 298 K under nitrogen atmosphere. To reaction mixture thus obtained, 2,7-diethoxynaphtharene (216 mg, 1.0 mmol) and methylene chloride (1.0 ml) were added. After the reaction mixture was stirred at r.t. for 24 h, it was poured into ice-cold water (20 ml). The aqueous layer was extracted with $CHCl_3$ (10 ml × 3). The combined extracts were washed with 2 *M* aqueous NaOH followed by washing with brine. The organic layers thus obtained were dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give cake. The crude product was purified by recrystallization from $CHCl_3$/methanol (1: 2 *v*/*v*) solution (81% yield). Colorless platelet single crystals suitable for X-ray diffraction were obtained by repeated crystallization from a $CHCl_3$/ methanol (1:2 *v*/*v*) solution (52% yield). Anal. Calcd

for $C_{28}H_{24}O_4$: C 79.22, H5.70. Found: C 79.32, H 5.84.; M.p. = 467–468 K. Spectroscopic data for the title compound are available in the archived CIF.

### S3. Refinement

All the H atoms were located in a difference Fourier map and were subsequently refined as riding atoms: C—H = 0.95 (aromatic), 0.98 (methyl) and 0.99 (methylene) Å, with $U_{iso}$(H) = 1.2 $U_{eq}$(C).

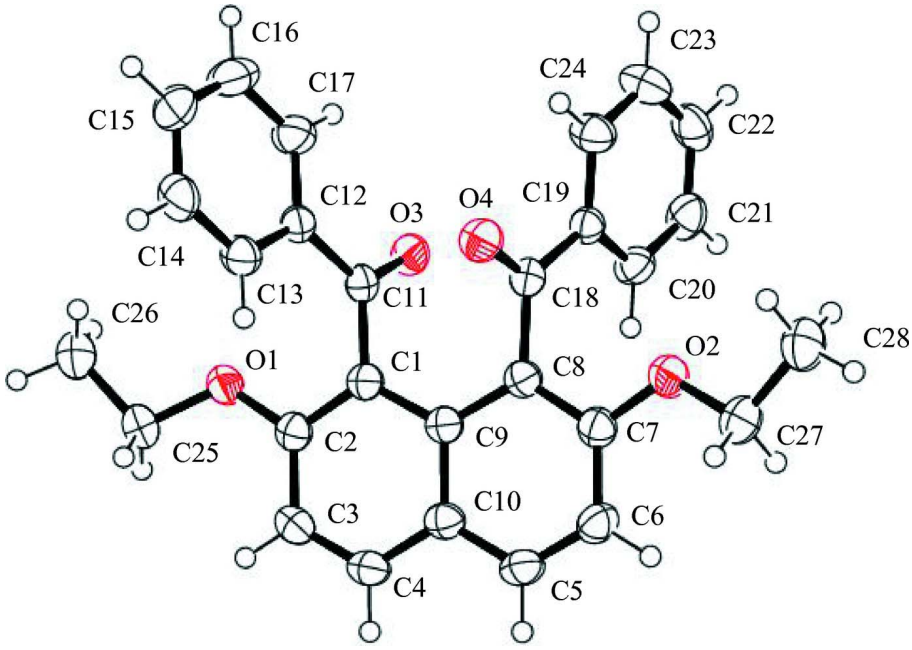


**Figure 1**

Molecular structure of the title molecule, with atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

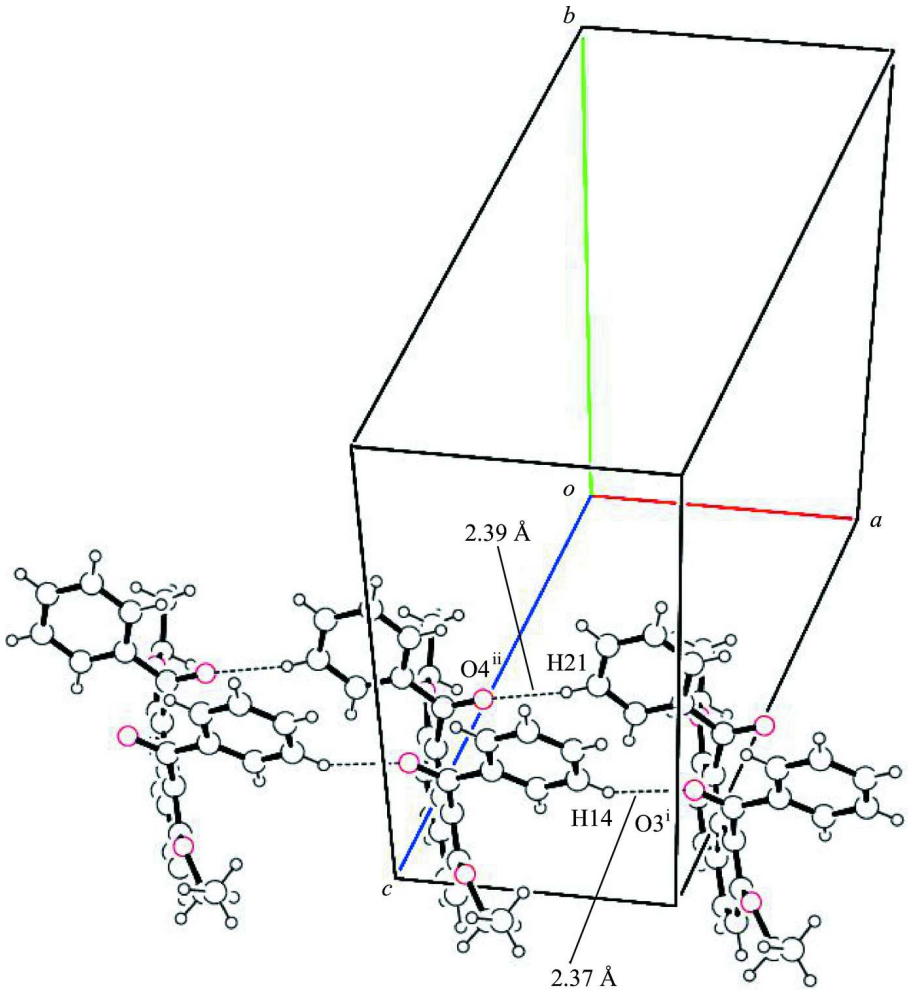


**Figure 2**

A partial view of the crystal packing of the title compound, showing the intermolecular C-H···O hydrogen bonds (see Table 1 for details; symmetry codes: (i) *x+1*, *y*, z; (ii) *x-1*, *y*, *z*).

## (8-Benzoyl-2,7-diethoxynaphthalen-1-yl)(phenyl)methanone

*Crystal data*

$C_{28}H_{24}O_4$
$M_r$ = 424.47
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 7.92185 (14) Å
$b$ = 20.6794 (4) Å
$c$ = 14.2130 (3) Å
$\beta$ = 106.043 (1)°
$V$ = 2237.68 (7) Å$^3$
$Z$ = 4
$F(000)$ = 896
$D_x$ = 1.260 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 37233 reflections
$\theta$ = 3.2–68.3°
$\mu$ = 0.67 mm$^{-1}$
$T$ = 193 K
Needle, colourless
0.60 × 0.50 × 0.10 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.689, $T_{max}$ = 0.936

39782 measured reflections
4076 independent reflections
3736 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041
$\theta_{max}$ = 68.2°, $\theta_{min}$ = 3.9°
$h$ = −9→9
$k$ = −24→24
$l$ = −17→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
$S$ = 1.05
4076 reflections
292 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.5163P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0075 (3)

*Special details*

**Experimental**. Spectroscopic data for the title compound: $^1$H NMR $\delta$ (300 MHz, $CDCl_3$); 0.91 (6*H*, t, *J* = 6.9 Hz), 3.95 (4*H*, q, *J* = 6.9 Hz), 7.16 (2*H*, d, *J* = 9.0 Hz), 7.35 (4*H*, t, *J* = 7.2 Hz), 7.49(2*H*, t, *J* = 7.2 Hz), 7.73 (4*H*, d, *J* = 7.2 Hz), 7.91 (2*H*, d, *J* = 9.0 Hz) p.p.m.; $^{13}$C NMR $\delta$ (75 MHz, $CDCl_3$); 13.92, 64.42, 111.89, 121.27, 125.05, 127.43, 128.54, 129.80, 131.79, 131.95, 138.93, 155.52, 197.04 p.p.m.; IR (KBr, cm$^{-1}$): 1665 (C═O), 1612, 1510, 1452 (Ar), 1266 (═C—O—C).

**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 | 0.29349 (11) | 0.08669 (4) | 1.04839 (6) | 0.0381 (2) |
| O2 | −0.21634 (12) | 0.01858 (4) | 0.55669 (6) | 0.0458 (2) |
| O3 | −0.02728 (10) | 0.16304 (4) | 0.86895 (6) | 0.0383 (2) |
| O4 | 0.08625 (10) | 0.12983 (4) | 0.66958 (6) | 0.0414 (2) |
| C1 | 0.12835 (13) | 0.06508 (5) | 0.88895 (8) | 0.0299 (2) |
| C2 | 0.21642 (14) | 0.04134 (5) | 0.98017 (8) | 0.0325 (3) |
| C3 | 0.21668 (15) | −0.02515 (6) | 1.00229 (9) | 0.0367 (3) |
| H3 | 0.2781 | −0.0406 | 1.0654 | 0.044* |
| C4 | 0.12768 (15) | −0.06697 (6) | 0.93199 (9) | 0.0375 (3) |
| H4 | 0.1268 | −0.1117 | 0.9471 | 0.045* |

| | | | | |
|---|---|---|---|---|
| C5 | −0.05370 (16) | −0.08988 (6) | 0.76527 (10) | 0.0398 (3) |
| H5 | −0.0536 | −0.1344 | 0.7817 | 0.048* |
| C6 | −0.14091 (16) | −0.07056 (6) | 0.67303 (9) | 0.0410 (3) |
| H6 | −0.2012 | −0.1012 | 0.6258 | 0.049* |
| C7 | −0.14038 (15) | −0.00448 (6) | 0.64861 (9) | 0.0361 (3) |
| C8 | −0.05794 (14) | 0.04132 (5) | 0.71690 (8) | 0.0314 (2) |
| C9 | 0.03474 (13) | 0.02192 (5) | 0.81381 (8) | 0.0302 (2) |
| C10 | 0.03651 (14) | −0.04555 (5) | 0.83711 (9) | 0.0341 (3) |
| C11 | 0.11515 (14) | 0.13756 (5) | 0.87770 (8) | 0.0299 (2) |
| C12 | 0.27340 (14) | 0.17695 (5) | 0.87977 (8) | 0.0320 (2) |
| C13 | 0.43562 (15) | 0.14857 (6) | 0.88777 (9) | 0.0390 (3) |
| H13 | 0.4468 | 0.1028 | 0.8910 | 0.047* |
| C14 | 0.58108 (16) | 0.18650 (7) | 0.89110 (10) | 0.0477 (3) |
| H14 | 0.6918 | 0.1668 | 0.8966 | 0.057* |
| C15 | 0.56497 (18) | 0.25260 (7) | 0.88645 (11) | 0.0542 (4) |
| H15 | 0.6650 | 0.2787 | 0.8893 | 0.065* |
| C16 | 0.4039 (2) | 0.28145 (7) | 0.87758 (13) | 0.0586 (4) |
| H16 | 0.3933 | 0.3272 | 0.8739 | 0.070* |
| C17 | 0.25816 (17) | 0.24361 (6) | 0.87402 (11) | 0.0455 (3) |
| H17 | 0.1474 | 0.2634 | 0.8676 | 0.055* |
| C18 | −0.05219 (14) | 0.10942 (5) | 0.67903 (8) | 0.0310 (2) |
| C19 | −0.21431 (14) | 0.14952 (5) | 0.65338 (8) | 0.0310 (2) |
| C20 | −0.37066 (15) | 0.12789 (6) | 0.66818 (9) | 0.0390 (3) |
| H20 | −0.3762 | 0.0860 | 0.6947 | 0.047* |
| C21 | −0.51890 (16) | 0.16678 (7) | 0.64465 (10) | 0.0459 (3) |
| H21 | −0.6257 | 0.1516 | 0.6547 | 0.055* |
| C22 | −0.51078 (18) | 0.22760 (7) | 0.60655 (10) | 0.0483 (3) |
| H22 | −0.6126 | 0.2541 | 0.5898 | 0.058* |
| C23 | −0.35529 (19) | 0.25015 (7) | 0.59266 (11) | 0.0531 (4) |
| H23 | −0.3498 | 0.2924 | 0.5673 | 0.064* |
| C24 | −0.20769 (17) | 0.21117 (6) | 0.61576 (10) | 0.0435 (3) |
| H24 | −0.1010 | 0.2267 | 0.6058 | 0.052* |
| C25 | 0.42914 (16) | 0.06661 (6) | 1.13328 (9) | 0.0420 (3) |
| H25A | 0.5139 | 0.0379 | 1.1140 | 0.050* |
| H25B | 0.3777 | 0.0429 | 1.1792 | 0.050* |
| C26 | 0.5191 (2) | 0.12683 (7) | 1.18056 (11) | 0.0571 (4) |
| H26A | 0.4357 | 0.1534 | 1.2032 | 0.068* |
| H26B | 0.5624 | 0.1513 | 1.1329 | 0.068* |
| H26C | 0.6181 | 0.1151 | 1.2365 | 0.068* |
| C27 | −0.26626 (18) | −0.02602 (7) | 0.47696 (9) | 0.0469 (3) |
| H27A | −0.3661 | −0.0530 | 0.4830 | 0.056* |
| H27B | −0.1667 | −0.0549 | 0.4766 | 0.056* |
| C28 | −0.31808 (19) | 0.01303 (7) | 0.38490 (10) | 0.0515 (3) |
| H28A | −0.2205 | 0.0413 | 0.3817 | 0.062* |
| H28B | −0.4211 | 0.0394 | 0.3844 | 0.062* |
| H28C | −0.3465 | −0.0160 | 0.3282 | 0.062* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0391 (4) | 0.0380 (4) | 0.0334 (4) | 0.0027 (3) | 0.0035 (3) | 0.0007 (3) |
| O2 | 0.0568 (6) | 0.0368 (5) | 0.0362 (5) | 0.0032 (4) | −0.0001 (4) | −0.0053 (4) |
| O3 | 0.0266 (4) | 0.0375 (4) | 0.0499 (5) | 0.0040 (3) | 0.0094 (4) | −0.0037 (4) |
| O4 | 0.0306 (4) | 0.0456 (5) | 0.0502 (5) | −0.0024 (3) | 0.0149 (4) | 0.0054 (4) |
| C1 | 0.0243 (5) | 0.0317 (6) | 0.0356 (6) | 0.0007 (4) | 0.0114 (4) | 0.0011 (4) |
| C2 | 0.0275 (5) | 0.0355 (6) | 0.0355 (6) | 0.0016 (4) | 0.0105 (5) | −0.0005 (5) |
| C3 | 0.0343 (6) | 0.0389 (6) | 0.0372 (6) | 0.0056 (5) | 0.0103 (5) | 0.0077 (5) |
| C4 | 0.0375 (6) | 0.0304 (6) | 0.0463 (7) | 0.0036 (5) | 0.0146 (5) | 0.0069 (5) |
| C5 | 0.0424 (7) | 0.0278 (6) | 0.0502 (7) | 0.0005 (5) | 0.0146 (6) | −0.0009 (5) |
| C6 | 0.0421 (7) | 0.0326 (6) | 0.0463 (7) | −0.0014 (5) | 0.0090 (6) | −0.0082 (5) |
| C7 | 0.0335 (6) | 0.0362 (6) | 0.0376 (6) | 0.0029 (5) | 0.0081 (5) | −0.0026 (5) |
| C8 | 0.0271 (5) | 0.0310 (6) | 0.0371 (6) | 0.0027 (4) | 0.0104 (5) | −0.0006 (5) |
| C9 | 0.0252 (5) | 0.0309 (5) | 0.0366 (6) | 0.0017 (4) | 0.0120 (4) | 0.0006 (4) |
| C10 | 0.0314 (6) | 0.0305 (6) | 0.0423 (7) | 0.0022 (4) | 0.0136 (5) | 0.0014 (5) |
| C11 | 0.0263 (5) | 0.0336 (6) | 0.0288 (5) | 0.0019 (4) | 0.0059 (4) | −0.0014 (4) |
| C12 | 0.0286 (6) | 0.0341 (6) | 0.0316 (6) | −0.0007 (4) | 0.0054 (4) | 0.0023 (4) |
| C13 | 0.0304 (6) | 0.0393 (6) | 0.0472 (7) | 0.0021 (5) | 0.0104 (5) | 0.0066 (5) |
| C14 | 0.0273 (6) | 0.0594 (8) | 0.0551 (8) | −0.0003 (5) | 0.0090 (6) | 0.0139 (7) |
| C15 | 0.0394 (7) | 0.0570 (9) | 0.0628 (9) | −0.0168 (6) | 0.0085 (6) | 0.0119 (7) |
| C16 | 0.0539 (8) | 0.0367 (7) | 0.0840 (11) | −0.0087 (6) | 0.0171 (8) | 0.0089 (7) |
| C17 | 0.0377 (7) | 0.0362 (7) | 0.0616 (8) | 0.0017 (5) | 0.0119 (6) | 0.0057 (6) |
| C18 | 0.0291 (5) | 0.0345 (6) | 0.0291 (6) | −0.0020 (4) | 0.0077 (4) | −0.0019 (4) |
| C19 | 0.0307 (6) | 0.0317 (6) | 0.0295 (6) | −0.0002 (4) | 0.0065 (4) | −0.0018 (4) |
| C20 | 0.0327 (6) | 0.0339 (6) | 0.0501 (7) | −0.0020 (5) | 0.0110 (5) | −0.0004 (5) |
| C21 | 0.0296 (6) | 0.0488 (7) | 0.0579 (8) | 0.0000 (5) | 0.0097 (6) | −0.0075 (6) |
| C22 | 0.0419 (7) | 0.0497 (8) | 0.0476 (8) | 0.0163 (6) | 0.0030 (6) | −0.0005 (6) |
| C23 | 0.0581 (8) | 0.0433 (7) | 0.0585 (9) | 0.0130 (6) | 0.0173 (7) | 0.0170 (6) |
| C24 | 0.0419 (7) | 0.0410 (7) | 0.0498 (8) | 0.0018 (5) | 0.0162 (6) | 0.0098 (6) |
| C25 | 0.0373 (6) | 0.0481 (7) | 0.0360 (6) | 0.0040 (5) | 0.0026 (5) | 0.0033 (5) |
| C26 | 0.0538 (8) | 0.0551 (9) | 0.0504 (8) | −0.0017 (7) | −0.0055 (7) | −0.0013 (7) |
| C27 | 0.0516 (8) | 0.0469 (7) | 0.0394 (7) | −0.0044 (6) | 0.0082 (6) | −0.0101 (6) |
| C28 | 0.0513 (8) | 0.0602 (9) | 0.0406 (7) | −0.0046 (6) | 0.0091 (6) | −0.0049 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.3657 (14) | C15—C16 | 1.382 (2) |
| O1—C25 | 1.4374 (14) | C15—H15 | 0.9500 |
| O2—C7 | 1.3645 (15) | C16—C17 | 1.3841 (18) |
| O2—C27 | 1.4297 (15) | C16—H16 | 0.9500 |
| O3—C11 | 1.2201 (13) | C17—H17 | 0.9500 |
| O4—C18 | 1.2167 (13) | C18—C19 | 1.4870 (15) |
| C1—C2 | 1.3815 (16) | C19—C20 | 1.3865 (16) |
| C1—C9 | 1.4315 (15) | C19—C24 | 1.3890 (17) |
| C1—C11 | 1.5079 (15) | C20—C21 | 1.3858 (17) |
| C2—C3 | 1.4104 (16) | C20—H20 | 0.9500 |

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|---|---|---|---|
| C3—C4 | 1.3616 (17) | C21—C22 | 1.378 (2) |
| C3—H3 | 0.9500 | C21—H21 | 0.9500 |
| C4—C10 | 1.4135 (17) | C22—C23 | 1.381 (2) |
| C4—H4 | 0.9500 | C22—H22 | 0.9500 |
| C5—C6 | 1.3620 (18) | C23—C24 | 1.3830 (18) |
| C5—C10 | 1.4108 (17) | C23—H23 | 0.9500 |
| C5—H5 | 0.9500 | C24—H24 | 0.9500 |
| C6—C7 | 1.4101 (17) | C25—C26 | 1.4985 (19) |
| C6—H6 | 0.9500 | C25—H25A | 0.9900 |
| C7—C8 | 1.3839 (16) | C25—H25B | 0.9900 |
| C8—C9 | 1.4277 (16) | C26—H26A | 0.9800 |
| C8—C18 | 1.5129 (15) | C26—H26B | 0.9800 |
| C9—C10 | 1.4331 (15) | C26—H26C | 0.9800 |
| C11—C12 | 1.4886 (15) | C27—C28 | 1.4952 (19) |
| C12—C17 | 1.3842 (17) | C27—H27A | 0.9900 |
| C12—C13 | 1.3886 (15) | C27—H27B | 0.9900 |
| C13—C14 | 1.3839 (17) | C28—H28A | 0.9800 |
| C13—H13 | 0.9500 | C28—H28B | 0.9800 |
| C14—C15 | 1.373 (2) | C28—H28C | 0.9800 |
| C14—H14 | 0.9500 | | |

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|---|---|---|---|
| C2—O1—C25 | 118.78 (9) | C17—C16—H16 | 120.0 |
| C7—O2—C27 | 119.05 (10) | C16—C17—C12 | 120.17 (12) |
| C2—C1—C9 | 120.12 (10) | C16—C17—H17 | 119.9 |
| C2—C1—C11 | 117.05 (10) | C12—C17—H17 | 119.9 |
| C9—C1—C11 | 122.29 (10) | O4—C18—C19 | 121.56 (10) |
| O1—C2—C1 | 115.67 (10) | O4—C18—C8 | 118.61 (10) |
| O1—C2—C3 | 122.57 (10) | C19—C18—C8 | 119.83 (9) |
| C1—C2—C3 | 121.66 (11) | C20—C19—C24 | 119.00 (11) |
| C4—C3—C2 | 119.15 (11) | C20—C19—C18 | 122.01 (10) |
| C4—C3—H3 | 120.4 | C24—C19—C18 | 118.96 (10) |
| C2—C3—H3 | 120.4 | C21—C20—C19 | 120.60 (11) |
| C3—C4—C10 | 121.70 (11) | C21—C20—H20 | 119.7 |
| C3—C4—H4 | 119.1 | C19—C20—H20 | 119.7 |
| C10—C4—H4 | 119.1 | C22—C21—C20 | 119.74 (12) |
| C6—C5—C10 | 121.79 (11) | C22—C21—H21 | 120.1 |
| C6—C5—H5 | 119.1 | C20—C21—H21 | 120.1 |
| C10—C5—H5 | 119.1 | C21—C22—C23 | 120.31 (12) |
| C5—C6—C7 | 119.08 (11) | C21—C22—H22 | 119.8 |
| C5—C6—H6 | 120.5 | C23—C22—H22 | 119.8 |
| C7—C6—H6 | 120.5 | C22—C23—C24 | 119.87 (12) |
| O2—C7—C8 | 115.47 (10) | C22—C23—H23 | 120.1 |
| O2—C7—C6 | 122.95 (11) | C24—C23—H23 | 120.1 |
| C8—C7—C6 | 121.56 (11) | C23—C24—C19 | 120.46 (12) |
| C7—C8—C9 | 120.10 (10) | C23—C24—H24 | 119.8 |
| C7—C8—C18 | 116.25 (10) | C19—C24—H24 | 119.8 |
| C9—C8—C18 | 123.15 (10) | O1—C25—C26 | 106.80 (10) |
| C8—C9—C1 | 124.53 (10) | O1—C25—H25A | 110.4 |

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| C8—C9—C10 | 117.78 (10) | C26—C25—H25A | 110.4 |
| C1—C9—C10 | 117.69 (10) | O1—C25—H25B | 110.4 |
| C5—C10—C4 | 120.66 (11) | C26—C25—H25B | 110.4 |
| C5—C10—C9 | 119.65 (11) | H25A—C25—H25B | 108.6 |
| C4—C10—C9 | 119.69 (11) | C25—C26—H26A | 109.5 |
| O3—C11—C12 | 121.04 (10) | C25—C26—H26B | 109.5 |
| O3—C11—C1 | 118.37 (9) | H26A—C26—H26B | 109.5 |
| C12—C11—C1 | 120.57 (9) | C25—C26—H26C | 109.5 |
| C17—C12—C13 | 119.30 (11) | H26A—C26—H26C | 109.5 |
| C17—C12—C11 | 118.99 (10) | H26B—C26—H26C | 109.5 |
| C13—C12—C11 | 121.71 (10) | O2—C27—C28 | 107.10 (11) |
| C14—C13—C12 | 120.42 (12) | O2—C27—H27A | 110.3 |
| C14—C13—H13 | 119.8 | C28—C27—H27A | 110.3 |
| C12—C13—H13 | 119.8 | O2—C27—H27B | 110.3 |
| C15—C14—C13 | 119.82 (12) | C28—C27—H27B | 110.3 |
| C15—C14—H14 | 120.1 | H27A—C27—H27B | 108.5 |
| C13—C14—H14 | 120.1 | C27—C28—H28A | 109.5 |
| C14—C15—C16 | 120.36 (12) | C27—C28—H28B | 109.5 |
| C14—C15—H15 | 119.8 | H28A—C28—H28B | 109.5 |
| C16—C15—H15 | 119.8 | C27—C28—H28C | 109.5 |
| C15—C16—C17 | 119.93 (13) | H28A—C28—H28C | 109.5 |
| C15—C16—H16 | 120.0 | H28B—C28—H28C | 109.5 |

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|---|---|---|---|
| C25—O1—C2—C1 | −161.30 (10) | C2—C1—C11—O3 | −108.29 (12) |
| C25—O1—C2—C3 | 22.47 (15) | C9—C1—C11—O3 | 63.28 (14) |
| C9—C1—C2—O1 | −176.52 (9) | C2—C1—C11—C12 | 70.21 (13) |
| C11—C1—C2—O1 | −4.75 (14) | C9—C1—C11—C12 | −118.21 (11) |
| C9—C1—C2—C3 | −0.25 (16) | O3—C11—C12—C17 | 1.58 (17) |
| C11—C1—C2—C3 | 171.51 (10) | C1—C11—C12—C17 | −176.88 (11) |
| O1—C2—C3—C4 | 175.92 (10) | O3—C11—C12—C13 | −178.72 (11) |
| C1—C2—C3—C4 | −0.08 (16) | C1—C11—C12—C13 | 2.81 (16) |
| C2—C3—C4—C10 | 0.64 (17) | C17—C12—C13—C14 | 0.74 (19) |
| C10—C5—C6—C7 | 0.25 (18) | C11—C12—C13—C14 | −178.95 (11) |
| C27—O2—C7—C8 | 164.97 (11) | C12—C13—C14—C15 | 0.0 (2) |
| C27—O2—C7—C6 | −13.53 (17) | C13—C14—C15—C16 | −0.6 (2) |
| C5—C6—C7—O2 | 176.45 (11) | C14—C15—C16—C17 | 0.4 (2) |
| C5—C6—C7—C8 | −1.97 (18) | C15—C16—C17—C12 | 0.3 (2) |
| O2—C7—C8—C9 | −176.28 (9) | C13—C12—C17—C16 | −0.9 (2) |
| C6—C7—C8—C9 | 2.25 (17) | C11—C12—C17—C16 | 178.82 (13) |
| O2—C7—C8—C18 | −4.12 (14) | C7—C8—C18—O4 | −105.71 (12) |
| C6—C7—C8—C18 | 174.41 (10) | C9—C8—C18—O4 | 66.19 (14) |
| C7—C8—C9—C1 | 178.17 (10) | C7—C8—C18—C19 | 73.69 (13) |
| C18—C8—C9—C1 | 6.58 (16) | C9—C8—C18—C19 | −114.42 (12) |
| C7—C8—C9—C10 | −0.83 (15) | O4—C18—C19—C20 | −176.90 (11) |
| C18—C8—C9—C10 | −172.42 (9) | C8—C18—C19—C20 | 3.72 (16) |
| C2—C1—C9—C8 | −178.97 (10) | O4—C18—C19—C24 | 1.44 (17) |
| C11—C1—C9—C8 | 9.71 (16) | C8—C18—C19—C24 | −177.94 (11) |
| C2—C1—C9—C10 | 0.03 (14) | C24—C19—C20—C21 | 0.84 (18) |

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|---|---|---|---|
| C11—C1—C9—C10 | −171.29 (9) | C18—C19—C20—C21 | 179.18 (11) |
| C6—C5—C10—C4 | −179.28 (11) | C19—C20—C21—C22 | −0.3 (2) |
| C6—C5—C10—C9 | 1.12 (17) | C20—C21—C22—C23 | −0.6 (2) |
| C3—C4—C10—C5 | 179.54 (11) | C21—C22—C23—C24 | 0.9 (2) |
| C3—C4—C10—C9 | −0.86 (17) | C22—C23—C24—C19 | −0.3 (2) |
| C8—C9—C10—C5 | −0.82 (15) | C20—C19—C24—C23 | −0.53 (19) |
| C1—C9—C10—C5 | −179.89 (10) | C18—C19—C24—C23 | −178.92 (12) |
| C8—C9—C10—C4 | 179.57 (10) | C2—O1—C25—C26 | 165.89 (11) |
| C1—C9—C10—C4 | 0.50 (15) | C7—O2—C27—C28 | −171.15 (11) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14···O3$^{i}$ | 0.95 | 2.37 | 3.2404 (16) | 153 |
| C21—H21···O4$^{ii}$ | 0.95 | 2.39 | 3.3326 (16) | 171 |

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