Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# [8-(4-Chlorobenzoyl)-2,7-dimethoxynaphthalen-1-yl](2,4,6-trimethylphenyl)methanone

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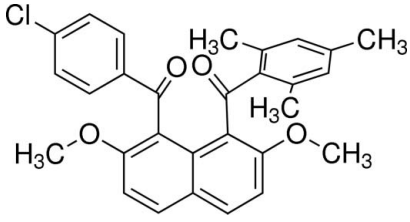
Received 14 February 2012; accepted 23 February 2012

Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.042; $wR$ factor = 0.131; data-to-parameter ratio = 13.6.

In the title compound, $C_{29}H_{25}ClO_4$, the dihedral angle between the benzene rings of the 2,4,6-trimethylbenzoyl group and the 4-chlorobenzoyl group is 65.19 (9)°. The dihedral angles between the naphthalene ring system and the benzene rings of the 2,4,6-trimethylbenzoyl group and the 4-chlorobenzoyl group are 85.66 (8) and 69.48 (8)°, respectively. In the crystal, two types of intermolecular C—H···O interactions and an intramolecular C—H···O interaction are observed. Moreover, there is a short intramolecular C═O···C═O contact of 2.614 (2) Å between the benzoyl substituents.

## Related literature

For electrophilic aromatic substitution of naphthalene derivatives, see: Okamoto & Yonezawa (2009); Okamoto *et al.* (2011). For the structures of closely related compounds, see: Mitsui *et al.* (2008); Muto *et al.* (2011*a*,*b*, 2012); Nakaema *et al.* (2007).




## Experimental

### Crystal data

$C_{29}H_{25}ClO_4$
$M_r$ = 472.94
Monoclinic, $P2_1/c$
$a$ = 11.6017 (2) Å
$b$ = 12.3381 (2) Å
$c$ = 16.2825 (3) Å
$\beta$ = 90.503 (1)°
$V$ = 2330.64 (7) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 1.73 mm$^{-1}$
$T$ = 193 K
0.30 × 0.20 × 0.10 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.625, $T_{max}$ = 0.846
40504 measured reflections
4266 independent reflections
3197 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.054

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.131
$S$ = 1.15
4266 reflections
313 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C23—H23···O2$^{i}$ | 0.95 | 2.54 | 3.413 (2) | 154 |
| C28—H28$A$···O1$^{ii}$ | 0.98 | 2.56 | 3.418 (3) | 147 |
| C29—H29$B$···O2 | 0.98 | 2.42 | 3.349 (3) | 157 |

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); 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 express their gratitude to Master Daichi Hijikata, Department of Organic and Polymer Materials Chemistry, Graduate School, Tokyo University of Agriculture and Technology, and Professor Keiichi Noguchi, Instrumentation Analysis Center, Tokyo University of Agriculture and Technology, for their technical advice.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2460).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o906 [doi:10.1107/S1600536812008112]

## [8-(4-Chlorobenzoyl)-2,7-dimethoxynaphthalen-1-yl](2,4,6-trimethylphenyl)-methanone

**Toyokazu Muto, Kosuke Sasagawa, Akiko Okamoto, Hideaki Oike and Noriyuki Yonezawa**

### Comment

In the course of our study on electrophilic aromatic aroylation of 2,7-dimethoxynaphthalene, *peri*-aroylnaphthalene compounds have proven to be formed regioselectively with the aid of suitable acidic mediators (Okamoto & Yonezawa, 2009; Okamoto, Mitsui *et al.*, 2011). Recently, we have reported the crystal structures of several 1,8-diaroylated naphthalene analogues exemplified by 1,8-bis(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (Nakaema *et al.*, 2007) and 1,8-bis(2,4,6-trimethylbenzoyl)-2,7-dimethoxynaphthalene (Muto *et al.*, 2012). The aroyl groups at the 1,8-positions of the naphthalene rings in these compounds are connected to the naphthalene rings in an almost perpendicular fashion. Besides, the crystal structures of 1-monoaroylated naphthalene derivatives and the *β*-isomers of 3-monoaroylated derivatives have been also clarified such as 1-(4-chlorobenzoyl)-2,7-dimethoxynaphthalene (Mitsui *et al.*, 2008), (2,7-dimethoxynaphthalen-1-yl)(2,4,6-trimethylphenyl)methanone (Muto *et al.*, 2011*a*) and (3,6-dimethoxynaphthalen-2-yl)(2,4,6-trimethylphenyl)methanone (Muto *et al.*, 2011*b*).

As a part of our continuing study on the molecular structures of these homologous molecules, the crystal structure of title compound, unsymmetrical *peri*-substituted naphthalene bearing 2,4,6-trimethylbenzoyl group and 4-chlorobenzoyl group, is discussed in this report.

The molecular structure of the title compound is displayed in Fig. 1. The 2,4,6-trimethylphenyl group and 4-chlorophenyl group are out of the plane of the naphthalene ring. Two kinds of phenyl rings make different dihedral angles with the naphthalene ring system, *i.e.*, the dihedral angle between the best planes of the 2,4,6-trimethylphenyl ring (C12—C17) and the naphthalene ring (C1—C10) is 85.66 (8)°, whereas, that between the best planes of the 4-chlorophenyl ring (C19—C24) and the naphthalene ring (C1—C10) is 69.48 (8)°. Each of dihedral angles is similar to that of the corresponding symmetric 1,8-diaroylnaphthalene. The dihedral angles between the best planes of the 2,4,6-trimethylphenyl rings and the naphthalene ring of 1,8-bis(2,4,6-trimethylbenzoyl)-2,7-dimethoxynaphthalene are 81.58 (5) and 84.92 (6)° (Muto *et al.*, 2012). In addition, the dihedral angles between the best planes of the 4-chlorophenyl rings and the naphthalene ring of 1,8-bis(4-chlorobenzoyl)-2,7-dimethoxynaphthalene are 71.55 (7) and 71.98 (7)° (Nakaema *et al.*, 2007).

Besides, an intramolecular C—H···O interaction between methyl group and carbonyl group is observed (C29—H29*b*···O2 = 2.42 Å; Fig. 1 and Table 1).

The crystal packing is additionally stabilized by an intermolecular C—H···O interaction between the oxygen atom (O2) of the carbonyl group and one hydrogen atom (H23) on 4-chlorophenyl group of the adjacent molecule along the *b* axis (C23—H23···O2$^{i}$; Fig. 2 and Table 1). Furthermore, an intermolecular C—H···O hydrogen bonding between the oxygen atom (O1) of the carbonyl group and one hydrogen atom (H28*a*) of the 4-methyl group on 2,4,6-trimethylphenyl ring of the adjacent molecule along the *b* axis is observed (C28—H28*a*···O1 $^{ii}$; Fig. 3 and Table 1).

## Experimental

To a 10 ml flask, 4-chlorobenzoyl chloride (0.40 mmol, 0.070 g), titanium chloride (1.20 mmol, 0.228 g) and methylene chloride (0.50 ml) were placed and stirred at rt. To the reaction mixture thus obtained, 1-(2,4,6-trimethylbenzoyl)-2,7-dimethoxynaphthalene (0.20 mmol, 0.067 g) was added. After the reaction mixture was stirred at rt for 9 h, it was poured into ice-cold water (10 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 (quant.). The crude product was purified by recrystallization from hexane and $CHCl_3$ (yield 2%).

$^1$H NMR $\delta$ (300 MHz, $CDCl_3$); 2.16 (6*H*, s), 2.25 (3*H*, s), 3.47 (3*H*, s), 3.68 (3*H*, s), 6.77 (2*H*, s), 7.10 (1*H*, d, *J* = 9.0 Hz), 7.23 (1*H*, d, *J* = 9.3 Hz), 7.34 (2*H*, d, *J* = 8.7 Hz), 7.74 (2*H*, d, *J* = 8.7 Hz), 7.92 (1*H*, d, *J* = 8.7 Hz), 7.94 (1*H*, d, *J* = 9.0 Hz) p.p.m..

$^{13}$C NMR $\delta$ (125 MHz, $CDCl_3$); 21.11, 21.35, 56.27, 56.83, 111.13, 112.39, 121.13, 124.87, 125.72, 128.13, 129.26, 129.57, 130.13, 132.43, 133.27, 137.88, 138.37, 138.57, 139.21, 157.20, 157.94, 195.83, 199.69 p.p.m..

IR (KBr); 1656 (C=O), 1607, 1514, 1457(Ar, naphthalene), 1271 (=C—O—C) $cm^{-1}$.

HRMS (*m/z*); $[M + Na]^+$ Calcd for $C_{29}H_{25}ClO_4Na$, 495.1370; found, 495.1339.

m.p. = 503.0–505.0 K.

## Refinement

All H atoms were found in a difference map 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).

## Computing details

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

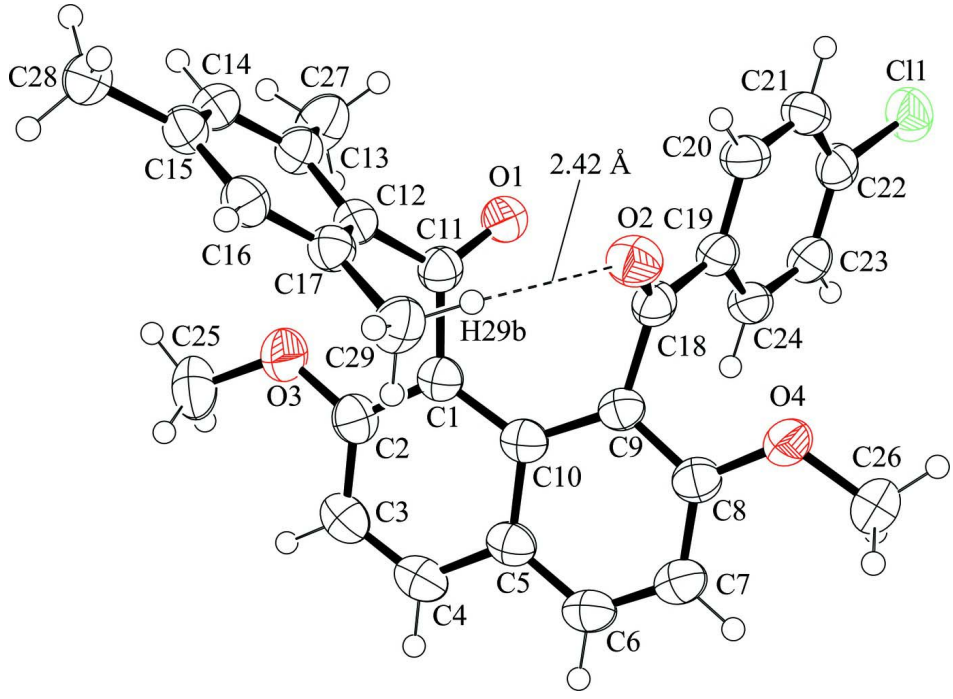


**Figure 1**

Molecular structure with displacement ellipsoids at 50% probability and a weak intramolecular C—H···O interactions.

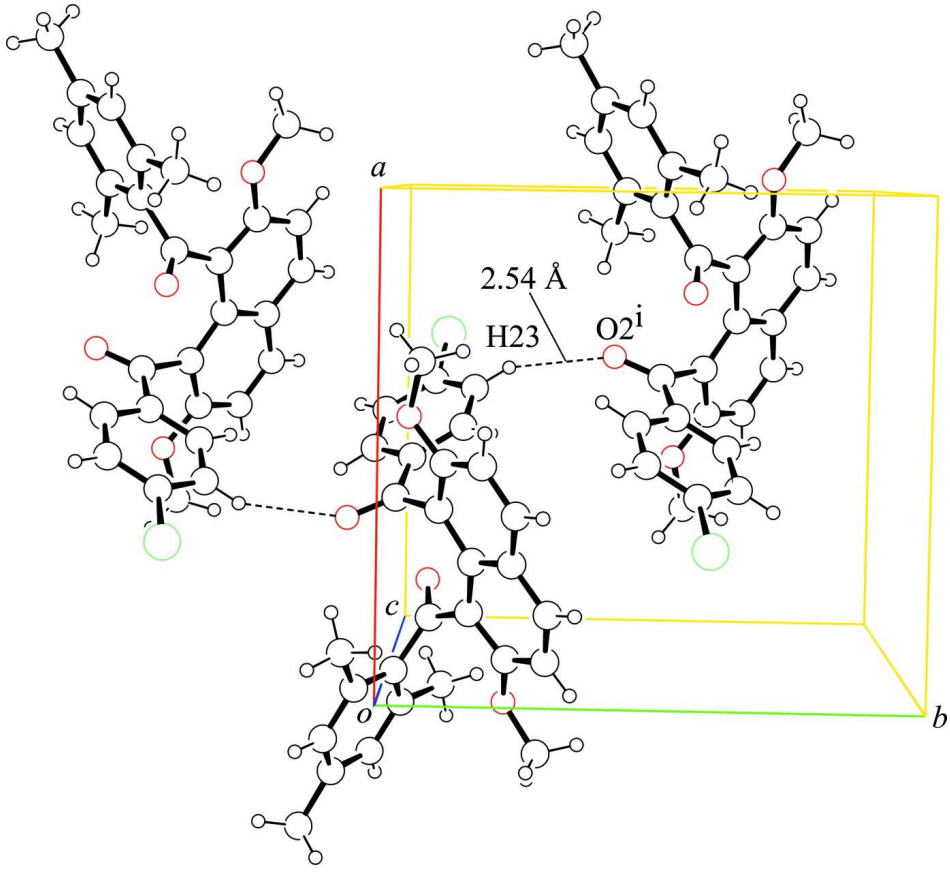


**Figure 2**

Intermolecular C23—H23···O2$^{i}$ interactions, viewed along the *c* axis [symmetry code: (i) –*x* + 1, *y* + 1/2, -*z* + 1/2].

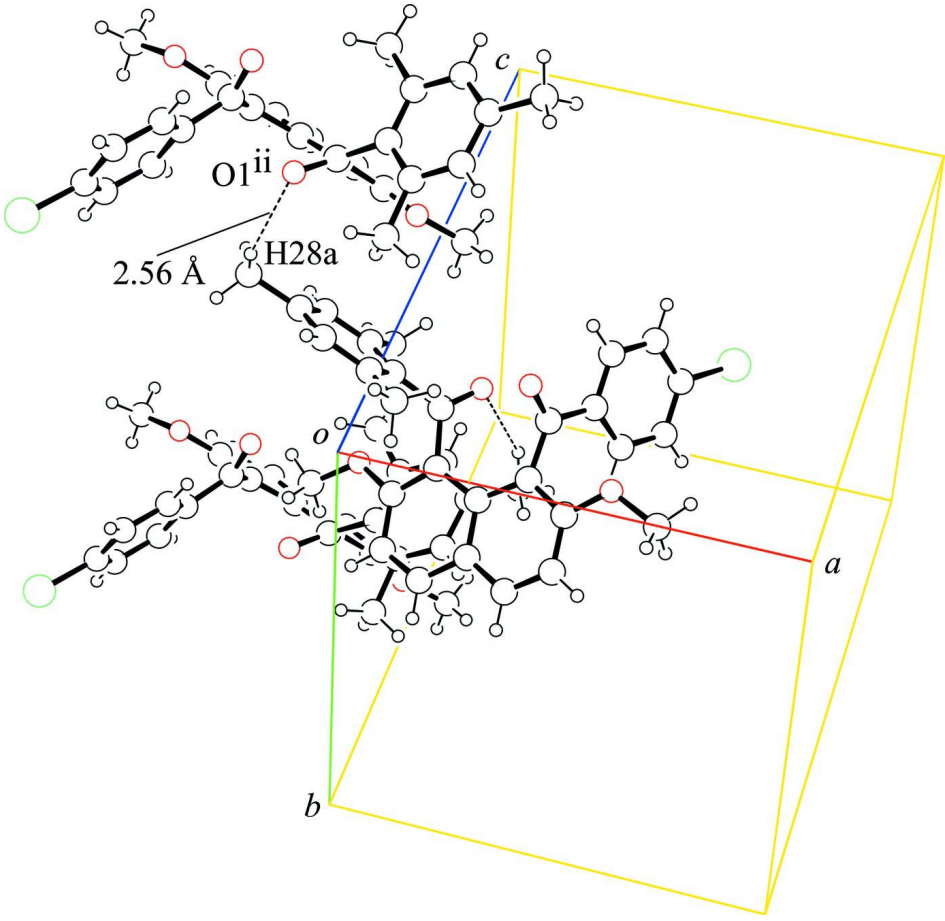


**Figure 3**

A packing diagram of the title compound, showing intermolecular C28—H28*a*···O1 $^{ii}$ interactions [symmetry code: (ii) –*x*, *y* - 1/2, -*z* + 1/2].

**[8-(4-Chlorobenzoyl)-2,7-dimethoxynaphthalen-1-yl](2,4,6- trimethylphenyl)methanone**

*Crystal data*

$C_{29}H_{25}ClO_4$
$M_r$ = 472.94
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 11.6017 (2) Å
$b$ = 12.3381 (2) Å
$c$ = 16.2825 (3) Å
$\beta$ = 90.503 (1)°
$V$ = 2330.64 (7) Å$^3$
$Z$ = 4
$F(000)$ = 992
$D_x$ = 1.348 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 30848 reflections
$\theta$ = 3.6–68.2°
$\mu$ = 1.73 mm$^{-1}$
$T$ = 193 K
Block, colorless
0.30 × 0.20 × 0.10 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)
$T_{min}$ = 0.625, $T_{max}$ = 0.846
40504 measured reflections

4266 independent reflections
3197 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 68.2°$, $\theta_{min} = 3.8°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -19 \rightarrow 19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.131$
$S = 1.15$
4266 reflections
313 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
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.4811P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0019 (2)

*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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.69235 (5) | 0.10716 (5) | 0.47771 (3) | 0.0588 (2) |
| O1 | 0.20277 (12) | 0.08172 (12) | 0.27478 (9) | 0.0481 (4) |
| O2 | 0.33992 (13) | −0.06714 (11) | 0.17961 (9) | 0.0507 (4) |
| O3 | −0.02500 (13) | 0.23149 (13) | 0.17441 (10) | 0.0584 (4) |
| O4 | 0.55327 (12) | 0.05151 (12) | 0.09221 (9) | 0.0492 (4) |
| C1 | 0.15846 (17) | 0.16470 (15) | 0.14692 (12) | 0.0393 (5) |
| C2 | 0.06994 (18) | 0.23511 (17) | 0.12574 (13) | 0.0454 (5) |
| C3 | 0.07890 (19) | 0.30891 (17) | 0.06063 (13) | 0.0489 (5) |
| H3 | 0.0164 | 0.3555 | 0.0470 | 0.059* |
| C4 | 0.17854 (19) | 0.31268 (17) | 0.01738 (13) | 0.0480 (5) |
| H4 | 0.1846 | 0.3618 | −0.0273 | 0.058* |
| C5 | 0.27304 (18) | 0.24567 (15) | 0.03717 (12) | 0.0411 (5) |
| C6 | 0.37374 (19) | 0.25238 (17) | −0.01035 (12) | 0.0453 (5) |
| H6 | 0.3761 | 0.3020 | −0.0549 | 0.054* |
| C7 | 0.46715 (18) | 0.19001 (17) | 0.00576 (12) | 0.0459 (5) |
| H7 | 0.5338 | 0.1948 | −0.0275 | 0.055* |
| C8 | 0.46389 (17) | 0.11829 (16) | 0.07222 (12) | 0.0416 (5) |
| C9 | 0.36732 (17) | 0.10735 (15) | 0.12160 (12) | 0.0379 (4) |
| C10 | 0.26600 (17) | 0.17072 (15) | 0.10417 (12) | 0.0382 (4) |
| C11 | 0.13588 (17) | 0.08802 (15) | 0.21667 (13) | 0.0402 (5) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.02723 (16) | 0.02178 (15) | 0.21450 (12) | 0.0389 (4) |
| C13 | −0.05043 (17) | 0.03204 (16) | 0.28009 (12) | 0.0424 (5) |
| C14 | −0.15272 (17) | −0.02578 (16) | 0.27704 (12) | 0.0436 (5) |
| H14 | −0.2059 | −0.0179 | 0.3207 | 0.052* |
| C15 | −0.18025 (17) | −0.09461 (16) | 0.21268 (13) | 0.0414 (5) |
| C16 | −0.10099 (17) | −0.10558 (16) | 0.14962 (13) | 0.0421 (5) |
| H16 | −0.1179 | −0.1536 | 0.1055 | 0.050* |
| C17 | 0.00221 (17) | −0.04836 (16) | 0.14928 (12) | 0.0404 (5) |
| C18 | 0.38091 (16) | 0.02322 (15) | 0.18818 (12) | 0.0394 (5) |
| C19 | 0.45487 (16) | 0.04930 (15) | 0.26164 (12) | 0.0388 (4) |
| C20 | 0.46449 (18) | −0.02682 (16) | 0.32407 (12) | 0.0449 (5) |
| H20 | 0.4207 | −0.0918 | 0.3211 | 0.054* |
| C21 | 0.53713 (18) | −0.00905 (17) | 0.39053 (13) | 0.0476 (5) |
| H21 | 0.5439 | −0.0617 | 0.4329 | 0.057* |
| C22 | 0.59999 (17) | 0.08638 (17) | 0.39460 (13) | 0.0446 (5) |
| C23 | 0.59021 (17) | 0.16476 (17) | 0.33401 (12) | 0.0442 (5) |
| H23 | 0.6331 | 0.2302 | 0.3376 | 0.053* |
| C24 | 0.51680 (17) | 0.14576 (16) | 0.26821 (12) | 0.0416 (5) |
| H24 | 0.5084 | 0.1994 | 0.2267 | 0.050* |
| C25 | −0.1238 (2) | 0.2937 (2) | 0.15477 (17) | 0.0650 (7) |
| H25A | −0.1048 | 0.3710 | 0.1584 | 0.078* |
| H25B | −0.1854 | 0.2768 | 0.1935 | 0.078* |
| H25C | −0.1496 | 0.2765 | 0.0988 | 0.078* |
| C26 | 0.66420 (18) | 0.0740 (2) | 0.05727 (15) | 0.0569 (6) |
| H26A | 0.6843 | 0.1501 | 0.0671 | 0.068* |
| H26B | 0.6614 | 0.0602 | −0.0020 | 0.068* |
| H26C | 0.7224 | 0.0272 | 0.0829 | 0.068* |
| C27 | −0.0277 (2) | 0.1066 (2) | 0.35151 (15) | 0.0607 (6) |
| H27A | −0.0005 | 0.1767 | 0.3312 | 0.073* |
| H27B | 0.0312 | 0.0746 | 0.3876 | 0.073* |
| H27C | −0.0990 | 0.1170 | 0.3823 | 0.073* |
| C28 | −0.29352 (17) | −0.15531 (17) | 0.21101 (14) | 0.0465 (5) |
| H28A | −0.2807 | −0.2295 | 0.1916 | 0.056* |
| H28B | −0.3477 | −0.1185 | 0.1739 | 0.056* |
| H28C | −0.3256 | −0.1573 | 0.2665 | 0.056* |
| C29 | 0.08631 (19) | −0.06858 (19) | 0.07974 (14) | 0.0513 (5) |
| H29A | 0.0572 | −0.1274 | 0.0448 | 0.062* |
| H29B | 0.1616 | −0.0889 | 0.1027 | 0.062* |
| H29C | 0.0942 | −0.0025 | 0.0469 | 0.062* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0508 (3) | 0.0793 (4) | 0.0463 (3) | 0.0077 (3) | −0.0052 (3) | −0.0046 (3) |
| O1 | 0.0416 (8) | 0.0590 (9) | 0.0436 (9) | −0.0052 (7) | −0.0041 (7) | 0.0070 (7) |
| O2 | 0.0572 (9) | 0.0425 (8) | 0.0525 (9) | −0.0051 (7) | −0.0018 (7) | 0.0016 (6) |
| O3 | 0.0450 (8) | 0.0610 (10) | 0.0693 (11) | 0.0130 (7) | 0.0078 (8) | 0.0100 (8) |
| O4 | 0.0459 (8) | 0.0531 (8) | 0.0487 (9) | 0.0059 (7) | 0.0125 (7) | 0.0070 (7) |
| C1 | 0.0413 (11) | 0.0385 (10) | 0.0381 (11) | −0.0018 (8) | −0.0014 (9) | −0.0014 (8) |
| C2 | 0.0439 (11) | 0.0458 (11) | 0.0464 (12) | 0.0012 (9) | −0.0032 (10) | −0.0028 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.0529 (13) | 0.0435 (11) | 0.0502 (13) | 0.0031 (10) | −0.0098 (10) | 0.0025 (9) |
| C4 | 0.0599 (14) | 0.0418 (11) | 0.0421 (12) | −0.0030 (10) | −0.0105 (10) | 0.0039 (9) |
| C5 | 0.0487 (11) | 0.0386 (10) | 0.0360 (11) | −0.0062 (9) | −0.0068 (9) | −0.0005 (8) |
| C6 | 0.0552 (13) | 0.0460 (11) | 0.0346 (11) | −0.0099 (10) | −0.0017 (9) | 0.0028 (9) |
| C7 | 0.0501 (12) | 0.0498 (12) | 0.0380 (11) | −0.0078 (10) | 0.0043 (9) | −0.0002 (9) |
| C8 | 0.0456 (11) | 0.0418 (11) | 0.0375 (11) | −0.0019 (9) | 0.0019 (9) | −0.0014 (8) |
| C9 | 0.0410 (10) | 0.0393 (10) | 0.0336 (10) | −0.0020 (8) | 0.0020 (8) | −0.0001 (8) |
| C10 | 0.0439 (11) | 0.0362 (10) | 0.0345 (10) | −0.0054 (8) | −0.0036 (8) | −0.0023 (8) |
| C11 | 0.0393 (11) | 0.0408 (10) | 0.0405 (12) | 0.0016 (8) | 0.0024 (9) | −0.0011 (8) |
| C12 | 0.0370 (10) | 0.0415 (10) | 0.0381 (11) | 0.0017 (8) | −0.0005 (8) | 0.0014 (8) |
| C13 | 0.0436 (11) | 0.0451 (11) | 0.0384 (11) | −0.0003 (9) | 0.0027 (9) | −0.0040 (9) |
| C14 | 0.0419 (11) | 0.0477 (11) | 0.0414 (11) | −0.0003 (9) | 0.0054 (9) | 0.0010 (9) |
| C15 | 0.0380 (11) | 0.0413 (11) | 0.0449 (12) | 0.0024 (8) | −0.0031 (9) | 0.0036 (9) |
| C16 | 0.0429 (11) | 0.0429 (11) | 0.0404 (11) | 0.0016 (9) | −0.0041 (9) | −0.0031 (9) |
| C17 | 0.0414 (11) | 0.0433 (10) | 0.0366 (11) | 0.0032 (8) | 0.0010 (9) | 0.0012 (8) |
| C18 | 0.0375 (10) | 0.0388 (10) | 0.0419 (11) | 0.0021 (8) | 0.0061 (9) | 0.0003 (8) |
| C19 | 0.0372 (10) | 0.0410 (10) | 0.0382 (11) | 0.0038 (8) | 0.0063 (8) | 0.0013 (8) |
| C20 | 0.0496 (12) | 0.0416 (11) | 0.0437 (12) | 0.0002 (9) | 0.0054 (10) | 0.0058 (9) |
| C21 | 0.0534 (12) | 0.0499 (12) | 0.0394 (12) | 0.0087 (10) | 0.0028 (10) | 0.0069 (9) |
| C22 | 0.0404 (11) | 0.0547 (12) | 0.0387 (12) | 0.0088 (9) | 0.0043 (9) | −0.0031 (9) |
| C23 | 0.0420 (11) | 0.0475 (11) | 0.0431 (12) | −0.0004 (9) | 0.0061 (9) | −0.0040 (9) |
| C24 | 0.0433 (11) | 0.0425 (11) | 0.0391 (11) | 0.0034 (9) | 0.0052 (9) | 0.0038 (9) |
| C25 | 0.0433 (13) | 0.0658 (15) | 0.0857 (19) | 0.0084 (11) | −0.0070 (12) | −0.0008 (13) |
| C26 | 0.0432 (12) | 0.0672 (15) | 0.0605 (15) | −0.0001 (10) | 0.0090 (11) | 0.0034 (11) |
| C27 | 0.0555 (14) | 0.0743 (16) | 0.0526 (14) | −0.0118 (12) | 0.0093 (11) | −0.0193 (12) |
| C28 | 0.0417 (11) | 0.0487 (12) | 0.0491 (13) | −0.0067 (9) | −0.0024 (9) | 0.0026 (9) |
| C29 | 0.0477 (12) | 0.0623 (14) | 0.0439 (13) | −0.0021 (10) | 0.0040 (10) | −0.0099 (10) |

*Geometric parameters (Å, º)*

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|---|---|---|---|
| Cl1—C22 | 1.738 (2) | C15—C16 | 1.391 (3) |
| O1—C11 | 1.221 (2) | C15—C28 | 1.513 (3) |
| O2—C18 | 1.220 (2) | C16—C17 | 1.390 (3) |
| O3—C2 | 1.363 (2) | C16—H16 | 0.9500 |
| O3—C25 | 1.414 (3) | C17—C29 | 1.522 (3) |
| O4—C8 | 1.362 (2) | C18—C19 | 1.501 (3) |
| O4—C26 | 1.439 (2) | C19—C20 | 1.388 (3) |
| C1—C2 | 1.386 (3) | C19—C24 | 1.394 (3) |
| C1—C10 | 1.436 (3) | C20—C21 | 1.383 (3) |
| C1—C11 | 1.503 (3) | C20—H20 | 0.9500 |
| C2—C3 | 1.402 (3) | C21—C22 | 1.386 (3) |
| C3—C4 | 1.360 (3) | C21—H21 | 0.9500 |
| C3—H3 | 0.9500 | C22—C23 | 1.385 (3) |
| C4—C5 | 1.408 (3) | C23—C24 | 1.383 (3) |
| C4—H4 | 0.9500 | C23—H23 | 0.9500 |
| C5—C6 | 1.409 (3) | C24—H24 | 0.9500 |
| C5—C10 | 1.433 (3) | C25—H25A | 0.9800 |
| C6—C7 | 1.353 (3) | C25—H25B | 0.9800 |
| C6—H6 | 0.9500 | C25—H25C | 0.9800 |
| C7—C8 | 1.399 (3) | C26—H26A | 0.9800 |

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|---|---|---|---|
| C7—H7 | 0.9500 | C26—H26B | 0.9800 |
| C8—C9 | 1.391 (3) | C26—H26C | 0.9800 |
| C9—C10 | 1.438 (3) | C27—H27A | 0.9800 |
| C9—C18 | 1.508 (3) | C27—H27B | 0.9800 |
| C11—C12 | 1.502 (3) | C27—H27C | 0.9800 |
| C12—C17 | 1.398 (3) | C28—H28A | 0.9800 |
| C12—C13 | 1.409 (3) | C28—H28B | 0.9800 |
| C13—C14 | 1.385 (3) | C28—H28C | 0.9800 |
| C13—C27 | 1.504 (3) | C29—H29A | 0.9800 |
| C14—C15 | 1.384 (3) | C29—H29B | 0.9800 |
| C14—H14 | 0.9500 | C29—H29C | 0.9800 |

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|---|---|---|---|
| C2—O3—C25 | 120.54 (18) | C12—C17—C29 | 122.43 (18) |
| C8—O4—C26 | 118.07 (16) | O2—C18—C19 | 120.45 (18) |
| C2—C1—C10 | 119.46 (18) | O2—C18—C9 | 120.58 (18) |
| C2—C1—C11 | 116.67 (17) | C19—C18—C9 | 118.73 (16) |
| C10—C1—C11 | 123.84 (17) | C20—C19—C24 | 118.92 (19) |
| O3—C2—C1 | 115.81 (18) | C20—C19—C18 | 118.71 (18) |
| O3—C2—C3 | 121.77 (19) | C24—C19—C18 | 122.35 (18) |
| C1—C2—C3 | 122.37 (19) | C21—C20—C19 | 120.7 (2) |
| C4—C3—C2 | 118.9 (2) | C21—C20—H20 | 119.7 |
| C4—C3—H3 | 120.6 | C19—C20—H20 | 119.7 |
| C2—C3—H3 | 120.6 | C20—C21—C22 | 119.26 (19) |
| C3—C4—C5 | 121.7 (2) | C20—C21—H21 | 120.4 |
| C3—C4—H4 | 119.2 | C22—C21—H21 | 120.4 |
| C5—C4—H4 | 119.2 | C23—C22—C21 | 121.3 (2) |
| C4—C5—C6 | 119.17 (19) | C23—C22—Cl1 | 119.84 (17) |
| C4—C5—C10 | 120.24 (18) | C21—C22—Cl1 | 118.89 (17) |
| C6—C5—C10 | 120.59 (19) | C24—C23—C22 | 118.64 (19) |
| C7—C6—C5 | 121.77 (19) | C24—C23—H23 | 120.7 |
| C7—C6—H6 | 119.1 | C22—C23—H23 | 120.7 |
| C5—C6—H6 | 119.1 | C23—C24—C19 | 121.19 (19) |
| C6—C7—C8 | 118.87 (19) | C23—C24—H24 | 119.4 |
| C6—C7—H7 | 120.6 | C19—C24—H24 | 119.4 |
| C8—C7—H7 | 120.6 | O3—C25—H25A | 109.5 |
| O4—C8—C9 | 114.75 (17) | O3—C25—H25B | 109.5 |
| O4—C8—C7 | 122.82 (17) | H25A—C25—H25B | 109.5 |
| C9—C8—C7 | 122.40 (19) | O3—C25—H25C | 109.5 |
| C8—C9—C10 | 119.62 (18) | H25A—C25—H25C | 109.5 |
| C8—C9—C18 | 113.73 (17) | H25B—C25—H25C | 109.5 |
| C10—C9—C18 | 126.60 (16) | O4—C26—H26A | 109.5 |
| C5—C10—C1 | 117.25 (18) | O4—C26—H26B | 109.5 |
| C5—C10—C9 | 116.69 (17) | H26A—C26—H26B | 109.5 |
| C1—C10—C9 | 126.05 (17) | O4—C26—H26C | 109.5 |
| O1—C11—C12 | 120.73 (17) | H26A—C26—H26C | 109.5 |
| O1—C11—C1 | 120.79 (18) | H26B—C26—H26C | 109.5 |
| C12—C11—C1 | 118.45 (18) | C13—C27—H27A | 109.5 |
| C17—C12—C13 | 120.09 (18) | C13—C27—H27B | 109.5 |
| C17—C12—C11 | 121.53 (17) | H27A—C27—H27B | 109.5 |

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| C13—C12—C11 | 118.38 (17) | C13—C27—H27C | 109.5 |
| C14—C13—C12 | 118.68 (18) | H27A—C27—H27C | 109.5 |
| C14—C13—C27 | 119.13 (18) | H27B—C27—H27C | 109.5 |
| C12—C13—C27 | 122.15 (18) | C15—C28—H28A | 109.5 |
| C15—C14—C13 | 122.35 (19) | C15—C28—H28B | 109.5 |
| C15—C14—H14 | 118.8 | H28A—C28—H28B | 109.5 |
| C13—C14—H14 | 118.8 | C15—C28—H28C | 109.5 |
| C14—C15—C16 | 117.96 (18) | H28A—C28—H28C | 109.5 |
| C14—C15—C28 | 120.80 (18) | H28B—C28—H28C | 109.5 |
| C16—C15—C28 | 121.23 (19) | C17—C29—H29A | 109.5 |
| C17—C16—C15 | 121.92 (19) | C17—C29—H29B | 109.5 |
| C17—C16—H16 | 119.0 | H29A—C29—H29B | 109.5 |
| C15—C16—H16 | 119.0 | C17—C29—H29C | 109.5 |
| C16—C17—C12 | 118.97 (18) | H29A—C29—H29C | 109.5 |
| C16—C17—C29 | 118.54 (18) | H29B—C29—H29C | 109.5 |

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| C25—O3—C2—C1 | −175.14 (19) | O1—C11—C12—C17 | −123.9 (2) |
| C25—O3—C2—C3 | 7.5 (3) | C1—C11—C12—C17 | 58.0 (3) |
| C10—C1—C2—O3 | −173.78 (17) | O1—C11—C12—C13 | 56.4 (3) |
| C11—C1—C2—O3 | 4.2 (3) | C1—C11—C12—C13 | −121.7 (2) |
| C10—C1—C2—C3 | 3.6 (3) | C17—C12—C13—C14 | −2.0 (3) |
| C11—C1—C2—C3 | −178.48 (19) | C11—C12—C13—C14 | 177.67 (18) |
| O3—C2—C3—C4 | 176.24 (19) | C17—C12—C13—C27 | −179.8 (2) |
| C1—C2—C3—C4 | −0.9 (3) | C11—C12—C13—C27 | −0.1 (3) |
| C2—C3—C4—C5 | −1.0 (3) | C12—C13—C14—C15 | 1.2 (3) |
| C3—C4—C5—C6 | 179.39 (19) | C27—C13—C14—C15 | 179.0 (2) |
| C3—C4—C5—C10 | 0.2 (3) | C13—C14—C15—C16 | 0.4 (3) |
| C4—C5—C6—C7 | −179.97 (19) | C13—C14—C15—C28 | −179.21 (19) |
| C10—C5—C6—C7 | −0.8 (3) | C14—C15—C16—C17 | −1.2 (3) |
| C5—C6—C7—C8 | −1.0 (3) | C28—C15—C16—C17 | 178.41 (19) |
| C26—O4—C8—C9 | −166.11 (18) | C15—C16—C17—C12 | 0.4 (3) |
| C26—O4—C8—C7 | 15.8 (3) | C15—C16—C17—C29 | 177.52 (19) |
| C6—C7—C8—O4 | 179.09 (18) | C13—C12—C17—C16 | 1.3 (3) |
| C6—C7—C8—C9 | 1.1 (3) | C11—C12—C17—C16 | −178.42 (18) |
| O4—C8—C9—C10 | −177.55 (17) | C13—C12—C17—C29 | −175.75 (19) |
| C7—C8—C9—C10 | 0.6 (3) | C11—C12—C17—C29 | 4.5 (3) |
| O4—C8—C9—C18 | 0.2 (3) | C8—C9—C18—O2 | −99.7 (2) |
| C7—C8—C9—C18 | 178.27 (18) | C10—C9—C18—O2 | 77.8 (3) |
| C4—C5—C10—C1 | 2.4 (3) | C8—C9—C18—C19 | 74.7 (2) |
| C6—C5—C10—C1 | −176.82 (18) | C10—C9—C18—C19 | −107.8 (2) |
| C4—C5—C10—C9 | −178.44 (18) | O2—C18—C19—C20 | −7.5 (3) |
| C6—C5—C10—C9 | 2.4 (3) | C9—C18—C19—C20 | 178.04 (17) |
| C2—C1—C10—C5 | −4.1 (3) | O2—C18—C19—C24 | 170.53 (18) |
| C11—C1—C10—C5 | 178.04 (17) | C9—C18—C19—C24 | −3.9 (3) |
| C2—C1—C10—C9 | 176.74 (18) | C24—C19—C20—C21 | −2.1 (3) |
| C11—C1—C10—C9 | −1.1 (3) | C18—C19—C20—C21 | 176.06 (17) |
| C8—C9—C10—C5 | −2.2 (3) | C19—C20—C21—C22 | 0.5 (3) |
| C18—C9—C10—C5 | −179.63 (18) | C20—C21—C22—C23 | 0.9 (3) |
| C8—C9—C10—C1 | 176.87 (18) | C20—C21—C22—Cl1 | −179.21 (15) |

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|---|---|---|---|
| C18—C9—C10—C1 | −0.5 (3) | C21—C22—C23—C24 | −0.6 (3) |
| C2—C1—C11—O1 | −127.5 (2) | Cl1—C22—C23—C24 | 179.47 (14) |
| C10—C1—C11—O1 | 50.4 (3) | C22—C23—C24—C19 | −1.0 (3) |
| C2—C1—C11—C12 | 50.6 (2) | C20—C19—C24—C23 | 2.3 (3) |
| C10—C1—C11—C12 | −131.50 (19) | C18—C19—C24—C23 | −175.70 (17) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C23—H23···O2$^{i}$ | 0.95 | 2.54 | 3.413 (2) | 154 |
| C28—H28*A*···O1$^{ii}$ | 0.98 | 2.56 | 3.418 (3) | 147 |
| C29—H29*B*···O2 | 0.98 | 2.42 | 3.349 (3) | 157 |

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