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# [2,7-Dihydroxy-8-(4-phenoxybenzoyl)-naphthalen-1-yl](4-phenoxyphenyl)-methanone

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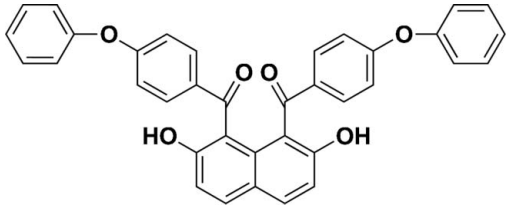
Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.037; $wR$ factor = 0.096; data-to-parameter ratio = 12.7.

In the title compound, $C_{36}H_{24}O_6$, the benzoyl groups at the 1- and 8-positions of the naphthalene system are in an *anti* orientation. Both carbonyl groups form intramolecular O—H···O hydrogen bonds with hydroxy groups affording six-membered rings. The benzene rings of the benzoyl groups make dihedral angles of 59.26 (13) and 59.09 (13)° with the naphthalene ring system. Zigzag C—H···O chains and ladder C—H···O chains between the phenoxybenzoyl groups along the *ab* diagonals form an undulating checkered sheet. The molecules are further connected into a three-dimensional network by C—H···$\pi$ interactions.

## Related literature

For electrophilic aromatic aroylation of the naphthalene core, see: Okamoto & Yonezawa (2009); Okamoto *et al.* (2011, 2013). For the structures of (2,7-dimethoxynaphthalene-1,8-diyl)bis(4-fluorophenyl)dimethanone and 2,7-dimethoxy-1,8-bis(4-phenoxybenzoyl)naphthalene, see: Watanabe *et al.* (2010) andr Hijikata *et al.* (2010), respectively.




## Experimental

### Crystal data

$C_{36}H_{24}O_6$
$M_r$ = 552.55
Monoclinic, *Cc*
$a$ = 16.0313 (3) Å
$b$ = 18.4956 (3) Å
$c$ = 12.1238 (2) Å
$\beta$ = 131.389 (1)°
$V$ = 2696.95 (9) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 0.75 mm$^{-1}$
$T$ = 193 K
0.60 × 0.55 × 0.10 mm

### Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min}$ = 0.661, $T_{max}$ = 0.929
22236 measured reflections
4868 independent reflections
4527 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
$S$ = 1.08
4868 reflections
382 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Absolute structure: Flack (1983), 2389 Friedel pairs
Flack parameter: 0.05 (19)

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

*Cg*1 and *Cg*2 are the centroids of the C25—C30 and C31—C36 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5*A*···O1 | 0.84 | 1.83 | 2.560 (3) | 145 |
| O6—H6*A*···O2 | 0.84 | 1.88 | 2.563 (3) | 138 |
| C26—H26···O4$^{i}$ | 0.95 | 2.48 | 3.377 (4) | 157 |
| C27—H27···O1$^{i}$ | 0.95 | 2.51 | 3.269 (4) | 137 |
| C32—H32···O3$^{ii}$ | 0.95 | 2.49 | 3.382 (4) | 156 |
| C33—H33···O2$^{ii}$ | 0.95 | 2.51 | 3.270 (4) | 137 |
| C14—H14···*Cg*1$^{iii}$ | 0.95 | 2.80 | 3.740 (2) | 171 |
| C21—H21···*Cg*2$^{iv}$ | 0.95 | 2.80 | 3.740 (2) | 171 |

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

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: RN2112).

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# supporting information

*Acta Cryst.* (2013). E**69**, o208–o209 [doi:10.1107/S1600536812052038]

# [2,7-Dihydroxy-8-(4-phenoxybenzoyl)naphthalen-1-yl](4-phenoxyphenyl)-methanone

**Daichi Hijikata, Kosuke Sasagawa, Sayaka Yoshiwaka, Akiko Okamoto and Noriyuki Yonezawa**

## S1. 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 *et al.*, 2011). As one of the applications of *peri*-aroylnaphthalene synthetic studies, the authors have integrated the resulting molecular unit to the poly(ether ketone) backbone *via* nucleophilic aromatic substitution polycondensation (Okamoto *et al.*, 2013). The poly(ether ketone)s composed of 1,8-diaroylenenaphthalene units show unique thermal properties and solubility for organic solvents. These notable properties could arise from the structural features of the 1,8-diaroylene naphthalene units. Under these circumstances, the authors have undertaken the X-ray crystal structural study of several 1,8-diaroylated naphthalene analogues exemplified by (2,7-dimethoxynaphthalene-1,8-diyl)bis(4-fluorophenyl)dimethanone (Watanabe *et al.*, 2010) and 2,7-dimethoxy-1,8-bis(4-phenoxybenzoyl)naphthalene (Hijikata *et al.*, 2010). These molecules have essentially the same non-coplanar features. The two aroyl groups are twisted so they are almost perpendicular to the naphthalene rings.

The molecular structure of the title compound is displayed in Fig. 1. Two benzoyl groups are on the 1,8-positions of the naphthalene ring and are in an *anti* orientation relative to one another. The benzene rings of the benzoyl groups make dihedral angles with the naphthalene ring of 59.26 (13) and 59.09 (13)°, respectively. The dihedral angles between the benzene rings of the benzoyl groups and those of the phenoxy groups are 69.05 (13) and 69.02 (13)°. Both carbonyl groups form intramolecular O—H···O hydrogen bonds with hydroxy groups affording six-membered rings. (Fig. 1, Table 1).

In the crystal structure, the molecular packing of the title compound is stabilized mainly by C—H···O and C—H···$\pi$ interactions. The aromatic hydrogen atoms of the phenoxy groups form two types of intermolecular C—H···O interactions with the ethereal oxygen atom of the phenoxy groups(C26—H26···O4$^{i}$= 2.48 Å, C32—H32···O3$^{ii}$= 2.49 Å; Fig. 2 and Table 1) and the carbonyl oxygen atom (C27—H27···O1$^{i}$= 2.51 Å, C33—H33···O2$^{ii}$= 2.51 Å; Fig. 2 and Table 1). Intermolecular C—H···$\pi$ interactions between the aromatic hydrogen atom of the benzoyl group and the centroid of the benzene ring of the phenoxy group (C14—H14···*Cg1*$^{iii}$= 2.80 Å, C21—H21···*Cg2*$^{iv}$= 2.80 Å; Fig. 3 and Table 1) are observed.

## S2. Experimental

To a stirring solution of 1,8-bis(4-phenoxybenzoyl)-2,7-dimethoxynaphthalene (1.0 mmol, 580 mg) in dichloromethane (1.0 ml) at 0°C was added 1.0 *M* boron tribromide solution in dichloromethane (4.4 ml) slowly, and the reaction mixture was allowed to reach the room temperature. After the reaction mixture had been stirred at room temperature for 48 h, the reaction mixture was cooled to 0 $^{o}$C and very slowly quenched with water and extracted with $CHCl_3$. The organic layer thus obtained was dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give a cake. The

crude product was purified by column chromatography (silica gel, $CHCl_3$) to give the title compound (isolated yield 88%). Single crystals suitable for X-ray diffraction were obtained by crystallization from $Et_2O$-hexane (*v*/*v* = 1:2).

$^1$H NMR $\delta$ (300 MHz, $CDCl_3$): 6.82–6.84 (4*H*, m), 7.08–7.26 (10*H*, m), 7.40 (4*H*, t, J=7.9 Hz) 7.86 (2*H*, d, J=8.9 Hz), 11.29 (2*H*, s) p.p.m.

$^{13}$C NMR $\delta$ (75 MHz, $CDCl_3$): 115.13, 117.03, 117.28, 120.02, 122.02, 124.46, 130.00, 130.68, 133.79, 136.09, 155.58, 161.74, 195.80 p.p.m.

IR (KBr): 3396(O—H), 1620 (C=O), 1608, 1583, 1487 (Ar, naphthalene) cm$^{-1}$.

HRMS (*m/z*): $[M + H]^+$ calcd for $C_{36}H_{25}O_6$, 553.1651 found, 553.1637.

m.p. 464.6–465.9 K.

## S3. Refinement

All the H atoms could be located in difference Fourier maps. All the H atoms were subsequently refined as riding atoms, with O5—H5A = 0.84, O6—H6A = 0.84, C—H = 0.95 (aromatic) Å, $U_{iso}$(H) = 1.2$U_{eq}$(O) and $U_{iso}$(H) = 1.2$U_{eq}$(C).

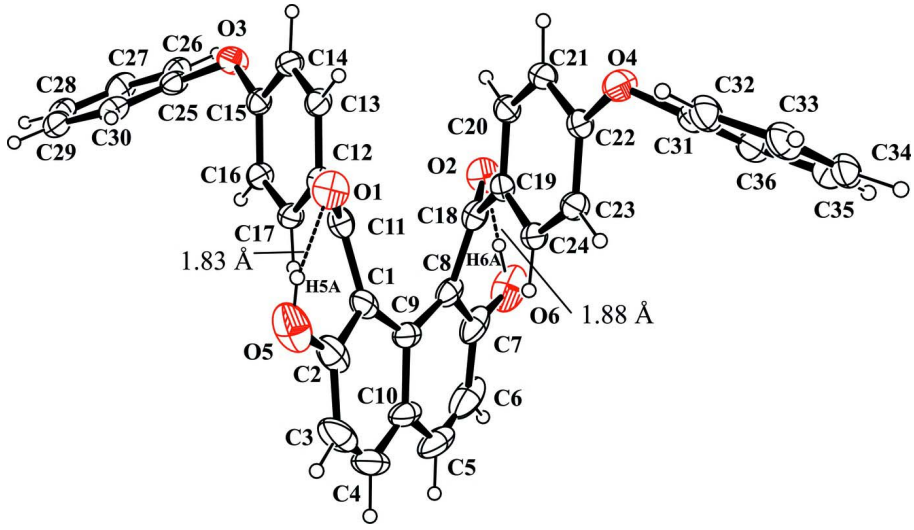


**Figure 1**

The molecular structure of title compound, showing 30% probability displacement ellipsoids. The intramolecular O—H···O hydrogen bond is shown as a dashed line.

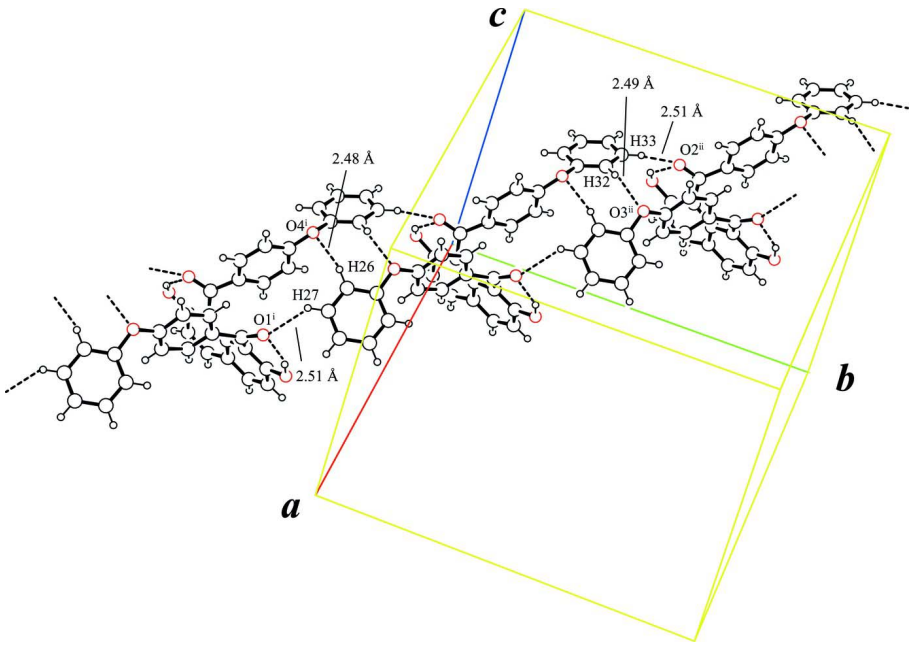


**Figure 2**

A partial crystal packing diagram of title compound. The intermolecular C—H···O interactions are shown as dashed lines.

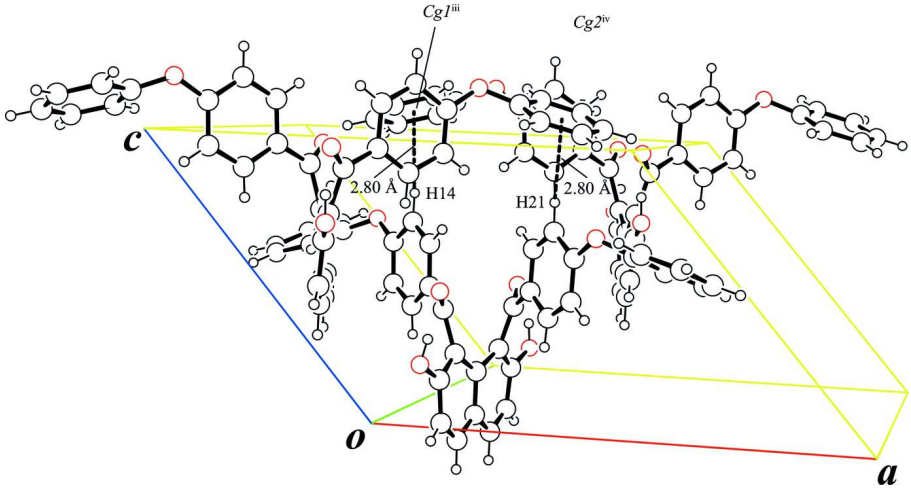


**Figure 3**

A partial crystal packing diagram of title compound. The intermolecular C—H···$\pi$ interactions are shown as dashed lines.

### [2,7-Dihydroxy-8-(4-phenoxybenzoyl)naphthalen-1-yl](4-phenoxyphenyl)methanone

*Crystal data*

$C_{36}H_{24}O_6$
$M_r$ = 552.55
Monoclinic, *Cc*
Hall symbol: C -2yc
$a$ = 16.0313 (3) Å
$b$ = 18.4956 (3) Å
$c$ = 12.1238 (2) Å
$\beta$ = 131.389 (1)°
$V$ = 2696.95 (9) Å$^3$
$Z$ = 4
$F(000)$ = 1152
$D_x$ = 1.361 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54187 Å
Cell parameters from 14515 reflections
$\theta$ = 4.4–68.1°
$\mu$ = 0.75 mm$^{-1}$

$T$ = 193 K
Block, yellow

0.60 × 0.55 × 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)
$T_{min}$ = 0.661, $T_{max}$ = 0.929

22236 measured reflections
4868 independent reflections
4527 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 68.1°, $\theta_{min}$ = 4.4°
$h$ = −19→19
$k$ = −22→22
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
$S$ = 1.08
4868 reflections
382 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 1.2872P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00222 (11)
Absolute structure: Flack (1983), 2389 Friedel pairs
Absolute structure parameter: 0.05 (19)

*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 | 0.46616 (15) | 0.24827 (8) | 0.4776 (2) | 0.0586 (5) |
| O2 | 0.36810 (15) | 0.00189 (8) | 0.4778 (2) | 0.0589 (5) |
| O3 | 0.78626 (13) | −0.00972 (9) | 0.69269 (17) | 0.0540 (4) |
| O4 | 0.26329 (13) | 0.25987 (9) | 0.69275 (17) | 0.0540 (4) |
| O5 | 0.3598 (2) | 0.31569 (12) | 0.2334 (3) | 0.0889 (7) |
| H5A | 0.4028 | 0.3117 | 0.3252 | 0.107* |
| O6 | 0.2301 (2) | −0.06560 (12) | 0.2333 (3) | 0.0887 (7) |
| H6A | 0.2881 | −0.0652 | 0.3226 | 0.106* |
| C1 | 0.33277 (19) | 0.18893 (13) | 0.2506 (2) | 0.0461 (5) |
| C2 | 0.3049 (3) | 0.25255 (17) | 0.1699 (3) | 0.0664 (8) |

| | | | | |
|---|---|---|---|---|
| C3 | 0.2213 (3) | 0.2516 (3) | 0.0165 (4) | 0.0985 (15) |
| H3 | 0.2020 | 0.2950 | −0.0374 | 0.118* |
| C4 | 0.1684 (3) | 0.1906 (3) | −0.0546 (3) | 0.1016 (16) |
| H4 | 0.1154 | 0.1909 | −0.1591 | 0.122* |
| C5 | 0.1337 (2) | 0.0591 (3) | −0.0547 (3) | 0.1027 (16) |
| H5 | 0.0822 | 0.0587 | −0.1592 | 0.123* |
| C6 | 0.1529 (3) | −0.0033 (3) | 0.0189 (4) | 0.1012 (15) |
| H6 | 0.1190 | −0.0471 | −0.0339 | 0.121* |
| C7 | 0.2216 (2) | −0.00274 (17) | 0.1702 (3) | 0.0661 (8) |
| C8 | 0.27453 (17) | 0.06111 (13) | 0.2507 (2) | 0.0460 (5) |
| C9 | 0.26698 (17) | 0.12502 (15) | 0.1775 (2) | 0.0487 (5) |
| C10 | 0.1889 (2) | 0.1250 (2) | 0.0211 (3) | 0.0757 (9) |
| C11 | 0.43775 (19) | 0.19208 (11) | 0.4067 (2) | 0.0426 (5) |
| C12 | 0.51884 (16) | 0.13161 (11) | 0.4748 (2) | 0.0369 (4) |
| C13 | 0.59891 (17) | 0.12824 (12) | 0.6275 (2) | 0.0422 (5) |
| H13 | 0.5936 | 0.1599 | 0.6843 | 0.051* |
| C14 | 0.68556 (17) | 0.07991 (13) | 0.6977 (2) | 0.0452 (5) |
| H14 | 0.7375 | 0.0764 | 0.8019 | 0.054* |
| C15 | 0.69610 (16) | 0.03646 (11) | 0.6145 (2) | 0.0399 (4) |
| C16 | 0.61693 (17) | 0.03743 (12) | 0.4626 (2) | 0.0413 (5) |
| H16 | 0.6240 | 0.0065 | 0.4066 | 0.050* |
| C17 | 0.52707 (17) | 0.08406 (12) | 0.3929 (2) | 0.0405 (5) |
| H17 | 0.4707 | 0.0836 | 0.2888 | 0.049* |
| C18 | 0.32565 (17) | 0.05816 (11) | 0.4067 (2) | 0.0427 (5) |
| C19 | 0.31261 (16) | 0.11833 (11) | 0.4748 (2) | 0.0368 (4) |
| C20 | 0.38516 (17) | 0.12173 (12) | 0.6276 (2) | 0.0421 (5) |
| H20 | 0.4471 | 0.0899 | 0.6844 | 0.050* |
| C21 | 0.36894 (18) | 0.17017 (13) | 0.6976 (2) | 0.0458 (5) |
| H21 | 0.4215 | 0.1738 | 0.8018 | 0.055* |
| C22 | 0.27547 (17) | 0.21371 (11) | 0.6151 (2) | 0.0399 (4) |
| C23 | 0.20247 (17) | 0.21294 (12) | 0.4623 (2) | 0.0412 (5) |
| H23 | 0.1397 | 0.2441 | 0.4063 | 0.049* |
| C24 | 0.22251 (17) | 0.16599 (12) | 0.3928 (2) | 0.0402 (5) |
| H24 | 0.1746 | 0.1662 | 0.2886 | 0.048* |
| C25 | 0.83934 (16) | −0.02601 (13) | 0.6401 (2) | 0.0449 (5) |
| C26 | 0.87387 (19) | −0.09629 (14) | 0.6575 (3) | 0.0530 (6) |
| H26 | 0.8565 | −0.1317 | 0.6964 | 0.064* |
| C27 | 0.9345 (2) | −0.11477 (15) | 0.6174 (3) | 0.0567 (6) |
| H27 | 0.9577 | −0.1634 | 0.6274 | 0.068* |
| C28 | 0.9614 (2) | −0.06375 (15) | 0.5636 (3) | 0.0553 (6) |
| H28 | 1.0036 | −0.0769 | 0.5372 | 0.066* |
| C29 | 0.92693 (19) | 0.00719 (15) | 0.5477 (3) | 0.0532 (6) |
| H29 | 0.9457 | 0.0428 | 0.5108 | 0.064* |
| C30 | 0.86529 (18) | 0.02621 (13) | 0.5855 (2) | 0.0480 (5) |
| H30 | 0.8410 | 0.0747 | 0.5740 | 0.058* |
| C31 | 0.15740 (18) | 0.27605 (13) | 0.6400 (2) | 0.0447 (5) |
| C32 | 0.1402 (2) | 0.34624 (14) | 0.6574 (3) | 0.0536 (6) |
| H32 | 0.1966 | 0.3815 | 0.6966 | 0.064* |

| | | | | |
|---|---|---|---|---|
| C33 | 0.0399 (2) | 0.36505 (15) | 0.6174 (3) | 0.0565 (6) |
| H33 | 0.0268 | 0.4137 | 0.6274 | 0.068* |
| C34 | −0.0414 (2) | 0.31361 (15) | 0.5632 (3) | 0.0553 (6) |
| H34 | −0.1098 | 0.3267 | 0.5370 | 0.066* |
| C35 | −0.0229 (2) | 0.24338 (15) | 0.5473 (3) | 0.0536 (6) |
| H35 | −0.0788 | 0.2079 | 0.5097 | 0.064* |
| C36 | 0.0773 (2) | 0.22397 (13) | 0.5859 (2) | 0.0480 (5) |
| H36 | 0.0902 | 0.1754 | 0.5751 | 0.058* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0674 (11) | 0.0398 (8) | 0.0723 (11) | 0.0032 (8) | 0.0477 (10) | −0.0045 (8) |
| O2 | 0.0679 (12) | 0.0406 (9) | 0.0727 (11) | 0.0090 (8) | 0.0484 (10) | 0.0052 (8) |
| O3 | 0.0489 (9) | 0.0693 (11) | 0.0468 (8) | 0.0229 (8) | 0.0329 (8) | 0.0118 (7) |
| O4 | 0.0441 (9) | 0.0691 (11) | 0.0456 (8) | 0.0075 (8) | 0.0284 (7) | −0.0114 (7) |
| O5 | 0.1121 (19) | 0.0656 (13) | 0.1298 (19) | 0.0416 (13) | 0.0974 (18) | 0.0511 (13) |
| O6 | 0.0801 (15) | 0.0650 (13) | 0.1276 (19) | −0.0250 (11) | 0.0715 (14) | −0.0503 (13) |
| C1 | 0.0473 (12) | 0.0584 (14) | 0.0462 (12) | 0.0211 (10) | 0.0368 (11) | 0.0146 (10) |
| C2 | 0.0748 (18) | 0.0779 (19) | 0.0782 (18) | 0.0407 (15) | 0.0640 (17) | 0.0389 (15) |
| C3 | 0.081 (2) | 0.161 (4) | 0.080 (2) | 0.077 (3) | 0.064 (2) | 0.080 (3) |
| C4 | 0.0517 (18) | 0.217 (5) | 0.0412 (15) | 0.054 (3) | 0.0328 (14) | 0.038 (2) |
| C5 | 0.0345 (14) | 0.219 (5) | 0.0422 (15) | 0.004 (2) | 0.0201 (13) | −0.042 (2) |
| C6 | 0.0496 (17) | 0.167 (4) | 0.082 (2) | −0.031 (2) | 0.0411 (18) | −0.083 (3) |
| C7 | 0.0424 (13) | 0.080 (2) | 0.0774 (18) | −0.0113 (13) | 0.0403 (14) | −0.0387 (15) |
| C8 | 0.0302 (10) | 0.0581 (14) | 0.0470 (12) | 0.0028 (9) | 0.0244 (9) | −0.0147 (10) |
| C9 | 0.0329 (11) | 0.0798 (15) | 0.0347 (10) | 0.0158 (11) | 0.0230 (9) | −0.0004 (11) |
| C10 | 0.0336 (12) | 0.159 (3) | 0.0319 (11) | 0.0234 (16) | 0.0206 (10) | −0.0003 (16) |
| C11 | 0.0501 (12) | 0.0401 (11) | 0.0516 (12) | 0.0021 (9) | 0.0396 (11) | 0.0009 (9) |
| C12 | 0.0329 (10) | 0.0386 (10) | 0.0411 (10) | −0.0010 (8) | 0.0252 (9) | −0.0009 (8) |
| C13 | 0.0372 (11) | 0.0491 (11) | 0.0418 (10) | −0.0025 (9) | 0.0269 (10) | −0.0094 (9) |
| C14 | 0.0351 (11) | 0.0609 (13) | 0.0352 (10) | 0.0035 (9) | 0.0213 (9) | −0.0013 (9) |
| C15 | 0.0341 (10) | 0.0437 (11) | 0.0426 (10) | 0.0057 (9) | 0.0257 (9) | 0.0040 (9) |
| C16 | 0.0405 (11) | 0.0445 (11) | 0.0421 (10) | 0.0030 (9) | 0.0287 (9) | −0.0050 (8) |
| C17 | 0.0346 (10) | 0.0504 (12) | 0.0362 (10) | 0.0050 (9) | 0.0232 (9) | 0.0013 (8) |
| C18 | 0.0355 (10) | 0.0386 (11) | 0.0523 (12) | 0.0013 (9) | 0.0283 (10) | −0.0003 (9) |
| C19 | 0.0377 (10) | 0.0384 (10) | 0.0410 (10) | 0.0008 (8) | 0.0289 (9) | 0.0017 (8) |
| C20 | 0.0385 (11) | 0.0489 (11) | 0.0418 (10) | 0.0101 (9) | 0.0278 (9) | 0.0092 (9) |
| C21 | 0.0400 (11) | 0.0627 (13) | 0.0346 (10) | 0.0052 (10) | 0.0247 (9) | 0.0016 (9) |
| C22 | 0.0392 (11) | 0.0460 (11) | 0.0404 (10) | 0.0016 (9) | 0.0289 (9) | −0.0025 (9) |
| C23 | 0.0379 (10) | 0.0450 (11) | 0.0405 (10) | 0.0104 (9) | 0.0259 (9) | 0.0054 (8) |
| C24 | 0.0350 (11) | 0.0509 (12) | 0.0360 (10) | 0.0034 (9) | 0.0241 (9) | −0.0008 (8) |
| C25 | 0.0310 (10) | 0.0608 (14) | 0.0350 (10) | 0.0065 (9) | 0.0184 (9) | −0.0036 (9) |
| C26 | 0.0390 (12) | 0.0598 (14) | 0.0540 (13) | 0.0056 (10) | 0.0281 (11) | −0.0001 (11) |
| C27 | 0.0425 (12) | 0.0593 (15) | 0.0600 (14) | 0.0066 (11) | 0.0303 (11) | −0.0098 (12) |
| C28 | 0.0370 (11) | 0.0753 (16) | 0.0529 (12) | −0.0033 (11) | 0.0294 (11) | −0.0173 (12) |
| C29 | 0.0395 (12) | 0.0713 (16) | 0.0431 (12) | −0.0088 (11) | 0.0248 (10) | −0.0100 (11) |
| C30 | 0.0368 (11) | 0.0516 (13) | 0.0411 (11) | 0.0009 (9) | 0.0196 (10) | −0.0073 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C31 | 0.0428 (11) | 0.0600 (14) | 0.0358 (10) | 0.0109 (10) | 0.0280 (10) | 0.0043 (9) |
| C32 | 0.0589 (15) | 0.0611 (14) | 0.0552 (13) | 0.0054 (11) | 0.0439 (13) | 0.0000 (11) |
| C33 | 0.0672 (16) | 0.0597 (15) | 0.0581 (14) | 0.0193 (13) | 0.0481 (13) | 0.0102 (11) |
| C34 | 0.0507 (13) | 0.0771 (17) | 0.0521 (12) | 0.0203 (13) | 0.0400 (11) | 0.0176 (12) |
| C35 | 0.0514 (14) | 0.0697 (16) | 0.0444 (12) | 0.0038 (12) | 0.0336 (11) | 0.0090 (11) |
| C36 | 0.0547 (13) | 0.0530 (13) | 0.0417 (11) | 0.0106 (10) | 0.0342 (11) | 0.0075 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C11 | 1.228 (3) | C16—H16 | 0.9500 |
| O2—C18 | 1.230 (3) | C17—H17 | 0.9500 |
| O3—C15 | 1.380 (2) | C18—C19 | 1.481 (3) |
| O3—C25 | 1.392 (3) | C19—C20 | 1.393 (3) |
| O4—C22 | 1.377 (2) | C19—C24 | 1.397 (3) |
| O4—C31 | 1.395 (3) | C20—C21 | 1.372 (3) |
| O5—C2 | 1.355 (4) | C20—H20 | 0.9500 |
| O5—H5A | 0.8400 | C21—C22 | 1.383 (3) |
| O6—C7 | 1.348 (4) | C21—H21 | 0.9500 |
| O6—H6A | 0.8400 | C22—C23 | 1.391 (3) |
| C1—C2 | 1.401 (3) | C23—C24 | 1.387 (3) |
| C1—C9 | 1.435 (4) | C23—H23 | 0.9500 |
| C1—C11 | 1.485 (3) | C24—H24 | 0.9500 |
| C2—C3 | 1.399 (5) | C25—C26 | 1.373 (3) |
| C3—C4 | 1.329 (6) | C25—C30 | 1.382 (3) |
| C3—H3 | 0.9500 | C26—C27 | 1.387 (4) |
| C4—C10 | 1.423 (6) | C26—H26 | 0.9500 |
| C4—H4 | 0.9500 | C27—C28 | 1.370 (4) |
| C5—C6 | 1.364 (6) | C27—H27 | 0.9500 |
| C5—C10 | 1.428 (6) | C28—C29 | 1.387 (4) |
| C5—H5 | 0.9500 | C28—H28 | 0.9500 |
| C6—C7 | 1.381 (5) | C29—C30 | 1.381 (3) |
| C6—H6 | 0.9500 | C29—H29 | 0.9500 |
| C7—C8 | 1.404 (3) | C30—H30 | 0.9500 |
| C8—C9 | 1.435 (4) | C31—C32 | 1.372 (3) |
| C8—C18 | 1.484 (3) | C31—C36 | 1.376 (3) |
| C9—C10 | 1.422 (3) | C32—C33 | 1.383 (4) |
| C11—C12 | 1.484 (3) | C32—H32 | 0.9500 |
| C12—C13 | 1.392 (3) | C33—C34 | 1.379 (4) |
| C12—C17 | 1.396 (3) | C33—H33 | 0.9500 |
| C13—C14 | 1.375 (3) | C34—C35 | 1.374 (4) |
| C13—H13 | 0.9500 | C34—H34 | 0.9500 |
| C14—C15 | 1.385 (3) | C35—C36 | 1.390 (3) |
| C14—H14 | 0.9500 | C35—H35 | 0.9500 |
| C15—C16 | 1.383 (3) | C36—H36 | 0.9500 |
| C16—C17 | 1.387 (3) | | |
| | | | |
| C15—O3—C25 | 119.93 (16) | O2—C18—C8 | 120.5 (2) |
| C22—O4—C31 | 119.97 (16) | C19—C18—C8 | 121.35 (18) |

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|---|---|---|---|
| C2—O5—H5A | 109.5 | C20—C19—C24 | 118.61 (18) |
| C7—O6—H6A | 109.5 | C20—C19—C18 | 118.29 (18) |
| C2—C1—C9 | 119.7 (2) | C24—C19—C18 | 122.58 (18) |
| C2—C1—C11 | 115.2 (2) | C21—C20—C19 | 121.27 (19) |
| C9—C1—C11 | 124.8 (2) | C21—C20—H20 | 119.4 |
| O5—C2—C3 | 117.3 (3) | C19—C20—H20 | 119.4 |
| O5—C2—C1 | 122.7 (3) | C20—C21—C22 | 119.41 (19) |
| C3—C2—C1 | 120.0 (3) | C20—C21—H21 | 120.3 |
| C4—C3—C2 | 120.9 (3) | C22—C21—H21 | 120.3 |
| C4—C3—H3 | 119.6 | O4—C22—C21 | 116.27 (18) |
| C2—C3—H3 | 119.6 | O4—C22—C23 | 122.82 (18) |
| C3—C4—C10 | 121.9 (3) | C21—C22—C23 | 120.80 (18) |
| C3—C4—H4 | 119.1 | C24—C23—C22 | 119.10 (19) |
| C10—C4—H4 | 119.1 | C24—C23—H23 | 120.4 |
| C6—C5—C10 | 121.7 (3) | C22—C23—H23 | 120.4 |
| C6—C5—H5 | 119.2 | C23—C24—C19 | 120.58 (18) |
| C10—C5—H5 | 119.2 | C23—C24—H24 | 119.7 |
| C5—C6—C7 | 120.0 (3) | C19—C24—H24 | 119.7 |
| C5—C6—H6 | 120.0 | C26—C25—C30 | 121.1 (2) |
| C7—C6—H6 | 120.0 | C26—C25—O3 | 116.2 (2) |
| O6—C7—C6 | 116.1 (3) | C30—C25—O3 | 122.5 (2) |
| O6—C7—C8 | 122.9 (3) | C25—C26—C27 | 119.0 (2) |
| C6—C7—C8 | 121.0 (4) | C25—C26—H26 | 120.5 |
| C7—C8—C9 | 119.8 (2) | C27—C26—H26 | 120.5 |
| C7—C8—C18 | 115.2 (2) | C28—C27—C26 | 120.8 (2) |
| C9—C8—C18 | 124.7 (2) | C28—C27—H27 | 119.6 |
| C10—C9—C8 | 117.7 (3) | C26—C27—H27 | 119.6 |
| C10—C9—C1 | 117.6 (3) | C27—C28—C29 | 119.7 (2) |
| C8—C9—C1 | 124.71 (18) | C27—C28—H28 | 120.2 |
| C9—C10—C4 | 119.0 (3) | C29—C28—H28 | 120.2 |
| C9—C10—C5 | 118.9 (3) | C30—C29—C28 | 120.2 (2) |
| C4—C10—C5 | 122.1 (3) | C30—C29—H29 | 119.9 |
| O1—C11—C12 | 117.7 (2) | C28—C29—H29 | 119.9 |
| O1—C11—C1 | 120.6 (2) | C29—C30—C25 | 119.3 (2) |
| C12—C11—C1 | 121.13 (18) | C29—C30—H30 | 120.4 |
| C13—C12—C17 | 118.70 (18) | C25—C30—H30 | 120.4 |
| C13—C12—C11 | 118.15 (18) | C32—C31—C36 | 121.1 (2) |
| C17—C12—C11 | 122.61 (18) | C32—C31—O4 | 116.1 (2) |
| C14—C13—C12 | 121.21 (19) | C36—C31—O4 | 122.6 (2) |
| C14—C13—H13 | 119.4 | C31—C32—C33 | 119.3 (2) |
| C12—C13—H13 | 119.4 | C31—C32—H32 | 120.3 |
| C13—C14—C15 | 119.11 (19) | C33—C32—H32 | 120.3 |
| C13—C14—H14 | 120.4 | C34—C33—C32 | 120.4 (2) |
| C15—C14—H14 | 120.4 | C34—C33—H33 | 119.8 |
| O3—C15—C16 | 123.03 (18) | C32—C33—H33 | 119.8 |
| O3—C15—C14 | 115.80 (18) | C35—C34—C33 | 119.7 (2) |
| C16—C15—C14 | 121.08 (18) | C35—C34—H34 | 120.1 |
| C15—C16—C17 | 119.23 (19) | C33—C34—H34 | 120.1 |

| | | | |
|---|---|---|---|
| C15—C16—H16 | 120.4 | C34—C35—C36 | 120.3 (2) |
| C17—C16—H16 | 120.4 | C34—C35—H35 | 119.8 |
| C16—C17—C12 | 120.47 (18) | C36—C35—H35 | 119.8 |
| C16—C17—H17 | 119.8 | C31—C36—C35 | 119.1 (2) |
| C12—C17—H17 | 119.8 | C31—C36—H36 | 120.5 |
| O2—C18—C19 | 117.5 (2) | C35—C36—H36 | 120.5 |
| | | | |
| C9—C1—C2—O5 | 176.4 (2) | O3—C15—C16—C17 | 178.0 (2) |
| C11—C1—C2—O5 | −9.4 (3) | C14—C15—C16—C17 | 1.6 (3) |
| C9—C1—C2—C3 | −7.1 (3) | C15—C16—C17—C12 | 2.6 (3) |
| C11—C1—C2—C3 | 167.1 (2) | C13—C12—C17—C16 | −3.8 (3) |
| O5—C2—C3—C4 | 175.7 (3) | C11—C12—C17—C16 | 167.6 (2) |
| C1—C2—C3—C4 | −1.0 (4) | C7—C8—C18—O2 | 34.8 (3) |
| C2—C3—C4—C10 | 4.1 (5) | C9—C8—C18—O2 | −151.6 (2) |
| C10—C5—C6—C7 | 4.3 (5) | C7—C8—C18—C19 | −135.9 (2) |
| C5—C6—C7—O6 | 175.6 (3) | C9—C8—C18—C19 | 37.8 (3) |
| C5—C6—C7—C8 | −1.4 (4) | O2—C18—C19—C20 | 26.1 (3) |
| O6—C7—C8—C9 | 176.5 (2) | C8—C18—C19—C20 | −163.0 (2) |
| C6—C7—C8—C9 | −6.7 (3) | O2—C18—C19—C24 | −145.5 (2) |
| O6—C7—C8—C18 | −9.5 (3) | C8—C18—C19—C24 | 25.5 (3) |
| C6—C7—C8—C18 | 167.4 (2) | C24—C19—C20—C21 | 0.5 (3) |
| C7—C8—C9—C10 | 11.6 (3) | C18—C19—C20—C21 | −171.4 (2) |
| C18—C8—C9—C10 | −161.8 (2) | C19—C20—C21—C22 | 3.6 (3) |
| C7—C8—C9—C1 | −168.3 (2) | C31—O4—C22—C21 | −146.0 (2) |
| C18—C8—C9—C1 | 18.3 (3) | C31—O4—C22—C23 | 37.7 (3) |
| C2—C1—C9—C10 | 11.7 (3) | C20—C21—C22—O4 | 178.6 (2) |
| C11—C1—C9—C10 | −161.8 (2) | C20—C21—C22—C23 | −5.0 (3) |
| C2—C1—C9—C8 | −168.4 (2) | O4—C22—C23—C24 | 178.3 (2) |
| C11—C1—C9—C8 | 18.0 (3) | C21—C22—C23—C24 | 2.1 (3) |
| C8—C9—C10—C4 | 171.4 (2) | C22—C23—C24—C19 | 2.1 (3) |
| C1—C9—C10—C4 | −8.7 (3) | C20—C19—C24—C23 | −3.5 (3) |
| C8—C9—C10—C5 | −8.7 (3) | C18—C19—C24—C23 | 168.1 (2) |
| C1—C9—C10—C5 | 171.2 (2) | C15—O3—C25—C26 | −140.9 (2) |
| C3—C4—C10—C9 | 0.9 (4) | C15—O3—C25—C30 | 44.7 (3) |
| C3—C4—C10—C5 | −178.9 (3) | C30—C25—C26—C27 | −0.8 (3) |
| C6—C5—C10—C9 | 0.9 (4) | O3—C25—C26—C27 | −175.3 (2) |
| C6—C5—C10—C4 | −179.2 (3) | C25—C26—C27—C28 | 1.1 (4) |
| C2—C1—C11—O1 | 34.9 (3) | C26—C27—C28—C29 | −0.5 (4) |
| C9—C1—C11—O1 | −151.3 (2) | C27—C28—C29—C30 | −0.3 (3) |
| C2—C1—C11—C12 | −136.1 (2) | C28—C29—C30—C25 | 0.5 (3) |
| C9—C1—C11—C12 | 37.7 (3) | C26—C25—C30—C29 | 0.0 (3) |
| O1—C11—C12—C13 | 26.0 (3) | O3—C25—C30—C29 | 174.1 (2) |
| C1—C11—C12—C13 | −162.8 (2) | C22—O4—C31—C32 | −141.1 (2) |
| O1—C11—C12—C17 | −145.5 (2) | C22—O4—C31—C36 | 44.7 (3) |
| C1—C11—C12—C17 | 25.8 (3) | C36—C31—C32—C33 | −1.2 (3) |
| C17—C12—C13—C14 | 0.8 (3) | O4—C31—C32—C33 | −175.38 (19) |
| C11—C12—C13—C14 | −171.0 (2) | C31—C32—C33—C34 | 1.2 (3) |
| C12—C13—C14—C15 | 3.3 (3) | C32—C33—C34—C35 | −0.7 (4) |

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|---|---|---|---|
| C25—O3—C15—C16 | 37.3 (3) | C33—C34—C35—C36 | 0.2 (3) |
| C25—O3—C15—C14 | −146.1 (2) | C32—C31—C36—C35 | 0.6 (3) |
| C13—C14—C15—O3 | 178.8 (2) | O4—C31—C36—C35 | 174.45 (19) |
| C13—C14—C15—C16 | −4.5 (3) | C34—C35—C36—C31 | −0.1 (3) |

*Hydrogen-bond geometry (Å, º)*

Cg1 and Cg2 are the centroids of the C25—C30 and C31—C36 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5*A*···O1 | 0.84 | 1.83 | 2.560 (3) | 145 |
| O6—H6*A*···O2 | 0.84 | 1.88 | 2.563 (3) | 138 |
| C26—H26···O4$^{i}$ | 0.95 | 2.48 | 3.377 (4) | 157 |
| C27—H27···O1$^{i}$ | 0.95 | 2.51 | 3.269 (4) | 137 |
| C32—H32···O3$^{ii}$ | 0.95 | 2.49 | 3.382 (4) | 156 |
| C33—H33···O2$^{ii}$ | 0.95 | 2.51 | 3.270 (4) | 137 |
| C14—H14···*Cg*1$^{iii}$ | 0.95 | 2.80 | 3.740 (2) | 171 |
| C21—H21···*Cg*2$^{iv}$ | 0.95 | 2.80 | 3.740 (2) | 171 |

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