

(4-Ethoxybenzoyl)[8-(4-ethoxybenzoyl)-2,7-dimethoxynaphthalen-1-yl]-methanone

Kosuke Sasagawa, Rei Sakamoto, Daichi Hijikata, Noriyuki Yonezawa and Akiko Okamoto*

Department of Organic and Polymer Materials Chemistry, Tokyo University of Agriculture & Technology, Koganei, Tokyo 184-8588, Japan

Correspondence e-mail: aokamoto@cc.tuat.ac.jp

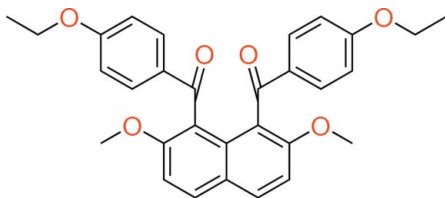
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Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.041; wR factor = 0.099; data-to-parameter ratio = 9.4.

The title molecule, $\text{C}_{30}\text{H}_{28}\text{O}_6$, possesses crystallographically imposed twofold symmetry, with two central C atoms in the naphthalene unit lying on the rotation axis along [001]. The 4-ethoxybenzoyl groups at the *peri* positions of the naphthalene ring system are disordered over two sets of sites with occupancies of 0.769 (4) and 0.231 (4). They are directed in opposite directions from the naphthalene plane (*anti* orientation). For the major component, the dihedral angle between the aroyl benzene ring and the naphthalene ring system is 75.62 (13)° [minor component 75.5 (4)°], and that between the aroyl benzene rings is 32.58 (15)°. In the crystal, molecules are linked *via* $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions, forming a three-dimensional network.

Related literature

For formation reactions of aroylated naphthalene compounds *via* electrophilic aromatic substitution of naphthalene derivatives, see: Okamoto & Yonezawa (2009); Okamoto *et al.* (2011). For the structures of closely related compounds, see: Hijikata *et al.* (2010); Sasagawa *et al.* (2011, 2012); Sasagawa, Sakamoto *et al.* (2013); Sasagawa, Takeuchi *et al.* (2013).



Experimental

Crystal data

$\text{C}_{30}\text{H}_{28}\text{O}_6$

$M_r = 484.52$

Orthorhombic, $Fddd$

$a = 19.6446$ (4) Å

$b = 21.5251$ (4) Å

$c = 22.9585$ (4) Å

$V = 9708.0$ (3) Å³

$Z = 16$

$\text{Cu } K\alpha$ radiation

$\mu = 0.75$ mm⁻¹

$T = 193$ K

$0.60 \times 0.50 \times 0.50$ mm

Data collection

Rigaku R-Axis RAPID diffractometer

Absorption correction: numerical (*NUMABS*; Higashi, 1999)

$T_{\min} = 0.662$, $T_{\max} = 0.706$

40441 measured reflections

2230 independent reflections

2113 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.099$

$S = 1.15$

2230 reflections

238 parameters

20 restraints

H-atom parameters constrained

$\Delta\rho_{\max} = 0.20$ e Å⁻³

$\Delta\rho_{\min} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the C8–C13 and C1–C6 rings, respectively

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C16}-\text{H16C}\cdots\text{O1}^i$	0.98	2.51	3.4919 (16)	175
$\text{C14}-\text{H14C}\cdots\text{Cg1}^i$	0.98	2.83	3.716 (2)	151
$\text{C15}-\text{H15B}\cdots\text{Cg2}^i$	0.99	2.80	3.6831 (19)	149

Symmetry code: (i) $-x + 1, y + \frac{1}{4}, z + \frac{1}{4}$

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (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: GK2562).

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supporting information

Acta Cryst. (2013). E69, o651 [https://doi.org/10.1107/S1600536813008581]

(4-Ethoxybenzoyl)[8-(4-ethoxybenzoyl)-2,7-dimethoxynaphthalen-1-yl]methanone

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S1. Comment

In the course of our study on selective electrophilic aromatic arylation of the naphthalene ring core, 1,8-diaroyl-naphthalene 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 structures of 1,8-diaroylated 2,7-dimethoxynaphthalene derivatives such as 1,8-bis(4-butoxybenzoyl)-2,7-dimethoxynaphthalene [$\{8-[4-(butoxy)benzoyl]-2,7-dimethoxynaphthalen-1-yl\}[4-(butoxy)phenyl]methanone$] (Sasagawa *et al.*, 2011), 1,8-bis(4-methoxybenzoyl)-2,7-dimethoxynaphthalene [$\{2,7-dimethoxy-8-(4-methoxybenzoyl)-naphthalen-1-yl\}(4-methoxyphenyl) methanone chloroform monosolvate$] (Sasagawa, Sakamoto *et al.*, 2013) and 1,8-bis(4-isobutylbenzoyl)-2,7-dimethoxynaphthalene [$\{2,7-dimethoxy-8-[4-(2-methylpropyl)benzoyl]-naphthalen-1-yl\}[4-(2-methylpropyl)phenyl-methanone\}$] (Sasagawa *et al.*, 2012).

The aryl groups in these compounds are almost perpendicularly attached to the naphthalene rings and oriented in opposite directions (*anti*-orientation). According to the authors' knowledge, most 1,8-diaroylnaphthalene derivatives have *anti*-oriented structures. Recently, we have also clarified another structure of the 1,8-diaroylnaphthalene derivatives, where the two aryl groups are situated in same direction (*syn*-orientation), 2,7-dimethoxy-1,8-bis(4-phenoxybenzoyl)-naphthalene (Hijikata *et al.*, 2010) and 1,8-bis(4-isopropoxybenzoyl)-2,7-dimethoxynaphthalene [$\{2,7-dimethoxy-8-[4-(propan-2-yloxy)benzoyl]naphthalen-1-yl\}[4-(propan-2-yloxy)phenyl]methanone$] (Sasagawa, Takeuchi *et al.*, 2013). 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, 1,8-diaroylated naphthalene bearing ethoxy groups on the aryl moieties, is discussed herein.

The molecular structure of the title compound is displayed in Fig 1. The title molecule lies on a crystallographic twofold axis so that the asymmetric unit contains one-half of the molecule. Thus, two 4-ethoxybenzoyl groups are situated in *anti*-orientation and are twisted away from the attached naphthalene ring. The dihedral angle between the best planes of the 4-ethoxyphenyl groups and the naphthalene ring system is 75.62 (13)° [or 75.5 (4)° for minor position].

The torsion angles along the bond between the naphthalene ring system and the bridging carbonyl moiety, C2—C1—C7—O1, is -109.85 (12)°, whereas that between the phenyl group and the bridging carbonyl moiety (O1—C7—C8—C9) is -169.0 (4)°.

In the crystal, C—H \cdots O and two kinds of C—H \cdots π interactions link the molecules and form a three-dimensional network (Table 1, Fig 2; symmetry code: $-x + 1, y + 1/4, z + 1/4$): a C—H \cdots O interaction between a hydrogen atom of the methyl moiety in the ethoxy group and the oxygen atom of the carbonyl moiety (C16—H16 \cdots O1 = 2.51 Å), a C—H \cdots π interaction between a hydrogen atom of the methoxy group and the π -system of benzene ring (C14—H14C \cdots Cg1 = 2.83 Å; Cg1 is the centroid of C8-C13 ring), and a C—H \cdots π interactions between a hydrogen atom of the methylene moiety in

the ethoxy group and the π -system of naphthalene ring ($C15-H15B \cdots Cg2 = 2.80 \text{ \AA}$; $Cg2$ is the centroid of C1-C6 ring).

S2. Experimental

The title compound was prepared by S_N2 reaction of 1,8-bis(4-hydroxybenzoyl)-2,7-dimethoxynaphthalene (2.0 mmol, 857 mg), which was obtained *via* S_NAr reaction of 1,8-bis(4-fluorobenzoyl)-2,7-dimethoxynaphthalene with sodium hydroxide, with ethyl iodide (6.0 mmol, 936 mg) and potassium carbonate (5.6 mmol, 774 mg) in *N,N*-dimethylformamide (DMF; 5.0 ml). After the reaction mixture was stirred at 328 K for 6 h, it was poured into water (30 ml) and the mixture was extracted with $CHCl_3$ (10 ml \times 3). The combined extracts were washed with brine. The organic layers thus obtained were dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give cake (93% yield). The crude product was purified by recrystallization from ethyl acetate (isolated yield 56%). Furthermore, the isolated product was crystallized from ethyl acetate to give single crystal. (m.p. = 473.6–477.6 K). Spectroscopic data for the title compound is available in the archived CIF.

S3. Refinement

All H atoms were found in a difference map and were subsequently refined as riding atoms, with $C-H = 0.95$ (aromatic), 0.98 (methyl) and 0.99 (methylene) \AA with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$. The 4-ethoxybenzene group is disordered over two positions with atoms C7/C7' and C16/C16' completely overlapping. The coordinates and anisotropic displacement parameters of these atomic pairs were constrained with EXYZ and EADP instructions of SHELXL-97 (Sheldrick, 2008). Moreover restraints were imposed on the geometry of the minor orientation with the instruction SAME. The occupancies of the two positions refined at 0.769 (4) and 0.231 (4).

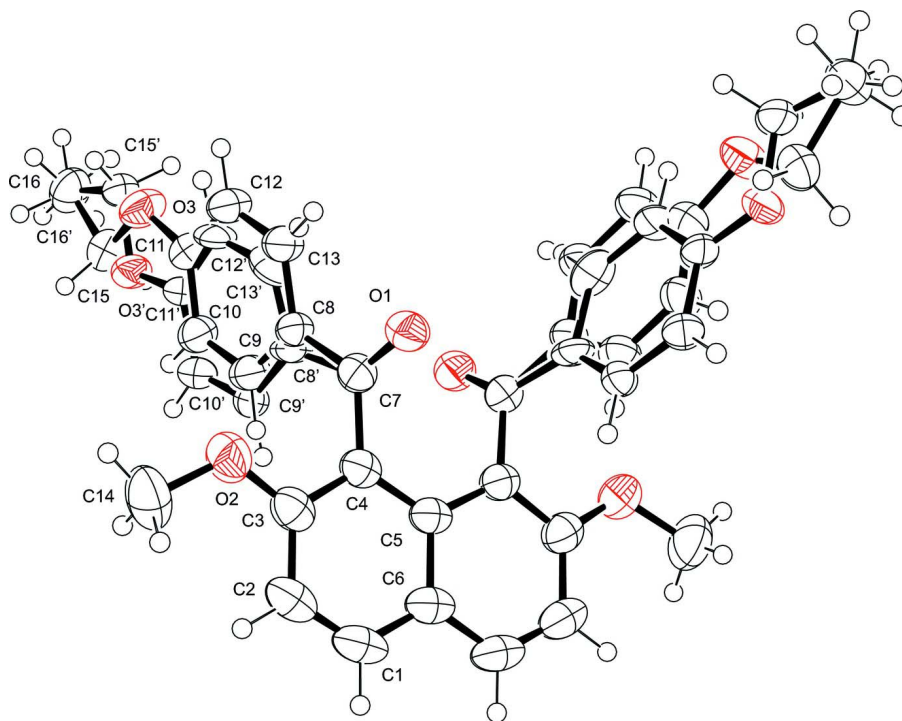


Figure 1

Molecular structure with displacement ellipsoids drawn at the 50% probability level. Symmetry code for generation of unlabeled atoms: $-x + 5/4, -y + 1/4, z$.

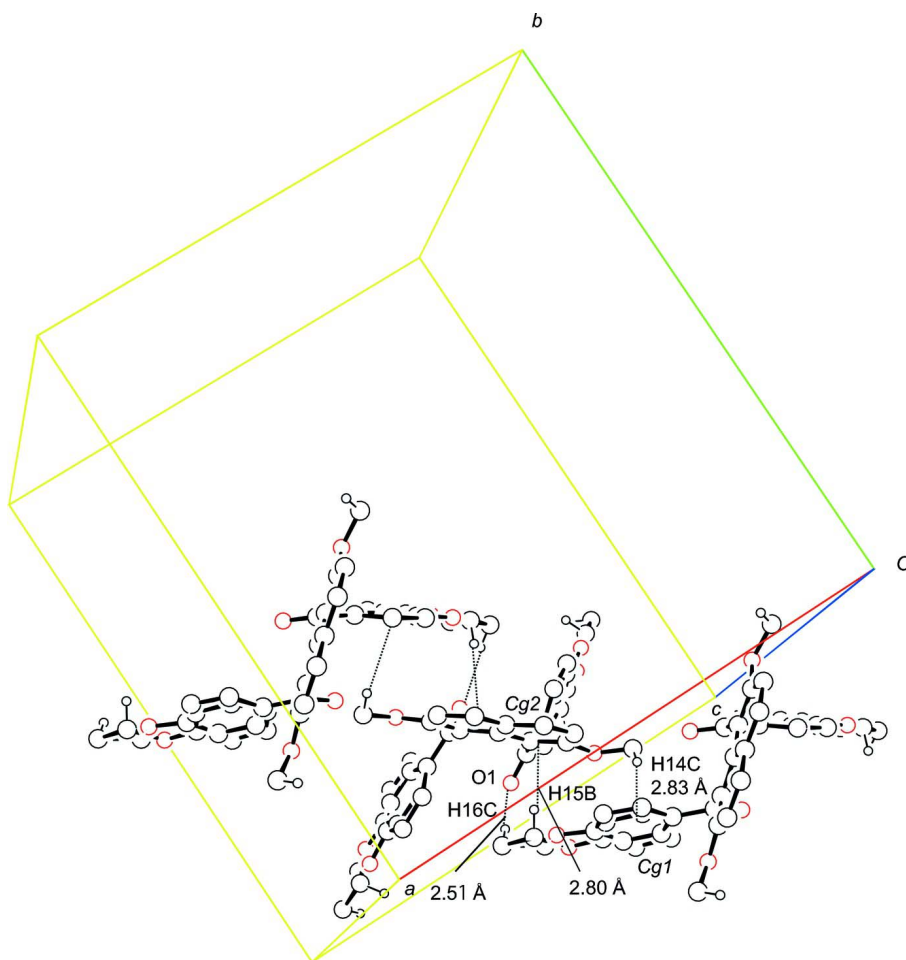


Figure 2

Intermolecular C—H...O interaction between the methyl group in the ethoxy group and the ketonic carbonyl group, C—H... π interaction between the methoxy group and the benzene ring, and that between the methylene moiety of the ethoxy group and the naphthalene group [symmetry code: $-x + 1, y + 1/4, z + 1/4$ along the a axis (dashed lines)].

(4-Ethoxybenzoyl)[8-(4-ethoxybenzoyl)-2,7-dimethoxynaphthalen-1-yl]methanone

Crystal data

$C_{30}H_{28}O_6$

$M_r = 484.52$

Orthorhombic, $Fddd$

Hall symbol: $-F\ 2uv\ 2vw$

$a = 19.6446(4)\ \text{\AA}$

$b = 21.5251(4)\ \text{\AA}$

$c = 22.9585(4)\ \text{\AA}$

$V = 9708.0(3)\ \text{\AA}^3$

$Z = 16$

$F(000) = 4096$

$D_x = 1.326\ \text{Mg m}^{-3}$

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

Cell parameters from 37043 reflections

$\theta = 3.6\text{--}68.2^\circ$

$\mu = 0.75\ \text{mm}^{-1}$

$T = 193\ \text{K}$

Block, colorless

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

*Data collection*Rigaku R-AXIS RAPID
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.000 pixels mm⁻¹ ω scansAbsorption correction: numerical
(NUMABS; Higashi, 1999) $T_{\min} = 0.662$, $T_{\max} = 0.706$

40441 measured reflections

2230 independent reflections

2113 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\max} = 68.2^\circ$, $\theta_{\min} = 3.6^\circ$ $h = -23 \rightarrow 23$ $k = -25 \rightarrow 25$ $l = -27 \rightarrow 27$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.041$ $wR(F^2) = 0.099$ $S = 1.15$

2230 reflections

238 parameters

20 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 5.2333P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$ *Special details*

Experimental. Spectroscopic Data: ¹H NMR δ (300 MHz, CDCl₃): 1.42 (6H, t, $J = 7.2$ Hz), 3.71 (6H, s), 4.05 (4H, q, $J = 7.2$ Hz), 6.78 (4H, br), 7.20 (2H, d, $J = 9.3$ Hz), 7.64 (4H, br), 7.92 (2H, d, $J = 9.3$ Hz) p.p.m. ¹³C NMR δ (75 MHz, CDCl₃): 14.7, 56.4, 63.4, 111.2, 113.5, 121.8, 125.6, 129.6, 131.3, 131.6, 131.9, 155.9, 162.4, 194.9 p.p.m. IR (KBr): 2980 (CH₃), 2938 (CH₂), 1658 (C=O), 1601, 1511, 1474 (Ar) cm⁻¹ HRMS (m/z): [M+H]⁺ calcd. for C₃₀H₂₉O₆, 485.1964, found, 485.1995

m.p. = 473.6—477.6 K

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.62404 (4)	0.04943 (4)	0.43348 (3)	0.0512 (2)	
O2	0.49033 (5)	0.01158 (5)	0.34362 (4)	0.0625 (3)	
C1	0.57676 (5)	0.08569 (5)	0.34527 (5)	0.0422 (3)	
C2	0.53143 (6)	0.05038 (6)	0.31286 (5)	0.0496 (3)	
C3	0.53080 (7)	0.05265 (6)	0.25136 (5)	0.0569 (3)	
H3	0.4983	0.0294	0.2297	0.068*	
C4	0.57734 (7)	0.08853 (6)	0.22388 (5)	0.0568 (4)	
H4	0.5778	0.0891	0.1825	0.068*	
C5	0.6250	0.1250	0.25413 (7)	0.0485 (4)	
C6	0.6250	0.1250	0.31675 (6)	0.0415 (3)	

C7	0.57588 (5)	0.07524 (5)	0.41049 (5)	0.0420 (3)	0.769 (4)
C8	0.5170 (3)	0.0946 (3)	0.4472 (2)	0.0379 (8)	0.769 (4)
C9	0.4673 (2)	0.1329 (2)	0.42483 (14)	0.0446 (7)	0.769 (4)
H9	0.4697	0.1451	0.3851	0.054*	0.769 (4)
C10	0.41390 (10)	0.15410 (10)	0.45895 (16)	0.0444 (6)	0.769 (4)
H10	0.3803	0.1809	0.4431	0.053*	0.769 (4)
C11	0.41044 (13)	0.13542 (12)	0.51683 (15)	0.0430 (5)	0.769 (4)
C12	0.45874 (12)	0.09486 (11)	0.53927 (10)	0.0485 (6)	0.769 (4)
H12	0.4552	0.0807	0.5784	0.058*	0.769 (4)
C13	0.51175 (15)	0.07517 (14)	0.50485 (12)	0.0431 (6)	0.769 (4)
H13	0.5452	0.0480	0.5206	0.052*	0.769 (4)
O3	0.36167 (7)	0.15461 (6)	0.55517 (6)	0.0562 (5)	0.769 (4)
C15	0.31301 (10)	0.19884 (8)	0.53540 (10)	0.0497 (5)	0.769 (4)
H15A	0.2836	0.1803	0.5050	0.060*	0.769 (4)
H15B	0.3362	0.2356	0.5188	0.060*	0.769 (4)
C16	0.27090 (7)	0.21734 (7)	0.58794 (6)	0.0606 (4)	0.769 (4)
H16A	0.2509	0.1801	0.6056	0.091*	0.769 (4)
H16B	0.2345	0.2456	0.5757	0.091*	0.769 (4)
H16C	0.3001	0.2382	0.6165	0.091*	0.769 (4)
C14	0.43576 (8)	-0.01802 (9)	0.31315 (8)	0.0809 (5)	
H14A	0.4035	0.0135	0.2994	0.121*	
H14B	0.4123	-0.0469	0.3394	0.121*	
H14C	0.4540	-0.0410	0.2797	0.121*	
C7'	0.57588 (5)	0.07524 (5)	0.41049 (5)	0.0420 (3)	0.231 (4)
C8'	0.5121 (10)	0.1018 (12)	0.4338 (7)	0.041 (3)	0.231 (4)
C9'	0.4664 (6)	0.1408 (6)	0.4061 (4)	0.036 (2)	0.231 (4)
H9'	0.4743	0.1505	0.3663	0.044*	0.231 (4)
C10'	0.4103 (3)	0.1663 (3)	0.4325 (4)	0.0432 (18)	0.231 (4)
H10'	0.3802	0.1926	0.4115	0.052*	0.231 (4)
C11'	0.3988 (3)	0.1525 (3)	0.4911 (5)	0.0376 (15)	0.231 (4)
C12'	0.4409 (4)	0.1139 (4)	0.5219 (3)	0.0413 (17)	0.231 (4)
H12'	0.4319	0.1036	0.5614	0.050*	0.231 (4)
C13'	0.4977 (5)	0.0900 (5)	0.4926 (5)	0.052 (3)	0.231 (4)
H13'	0.5283	0.0644	0.5138	0.062*	0.231 (4)
O3'	0.3435 (2)	0.1813 (2)	0.51409 (16)	0.0478 (14)	0.231 (4)
C15'	0.3322 (3)	0.1786 (3)	0.5762 (2)	0.0493 (17)	0.231 (4)
H15C	0.3721	0.1951	0.5974	0.059*	0.231 (4)
H15D	0.3243	0.1352	0.5887	0.059*	0.231 (4)
C16'	0.27090 (7)	0.21734 (7)	0.58794 (6)	0.0606 (4)	0.231 (4)
H16D	0.2791	0.2599	0.5746	0.091*	0.231 (4)
H16E	0.2615	0.2176	0.6299	0.091*	0.231 (4)
H16F	0.2317	0.2000	0.5671	0.091*	0.231 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0481 (5)	0.0616 (5)	0.0440 (4)	0.0054 (4)	-0.0029 (3)	0.0054 (4)
O2	0.0588 (5)	0.0675 (6)	0.0612 (6)	-0.0130 (4)	-0.0051 (4)	-0.0125 (4)

C1	0.0418 (6)	0.0462 (6)	0.0386 (6)	0.0093 (5)	-0.0023 (4)	-0.0038 (4)
C2	0.0472 (6)	0.0521 (7)	0.0493 (6)	0.0085 (5)	-0.0060 (5)	-0.0076 (5)
C3	0.0621 (7)	0.0598 (8)	0.0489 (7)	0.0135 (6)	-0.0169 (6)	-0.0130 (6)
C4	0.0716 (8)	0.0628 (8)	0.0360 (6)	0.0231 (7)	-0.0098 (6)	-0.0067 (5)
C5	0.0581 (9)	0.0513 (9)	0.0361 (8)	0.0211 (8)	0.000	0.000
C6	0.0434 (8)	0.0459 (8)	0.0351 (7)	0.0150 (6)	0.000	0.000
C7	0.0422 (6)	0.0431 (6)	0.0407 (6)	-0.0017 (4)	-0.0011 (4)	-0.0020 (4)
C8	0.0411 (12)	0.0420 (16)	0.031 (2)	-0.0027 (8)	0.0015 (16)	-0.0001 (18)
C9	0.0487 (13)	0.0489 (14)	0.0362 (16)	0.0005 (10)	0.0024 (13)	0.0050 (13)
C10	0.0433 (10)	0.0475 (10)	0.0423 (17)	0.0031 (7)	0.0035 (12)	0.0059 (12)
C11	0.0444 (13)	0.0454 (13)	0.0393 (14)	-0.0013 (9)	0.0061 (13)	0.0015 (11)
C12	0.0561 (12)	0.0547 (12)	0.0347 (11)	0.0017 (9)	0.0047 (9)	0.0065 (8)
C13	0.0458 (12)	0.0476 (14)	0.0361 (12)	0.0041 (9)	-0.0003 (9)	0.0026 (9)
O3	0.0581 (8)	0.0647 (8)	0.0458 (8)	0.0093 (6)	0.0157 (6)	0.0059 (6)
C15	0.0448 (10)	0.0496 (9)	0.0546 (11)	-0.0029 (7)	0.0069 (9)	0.0007 (8)
C16	0.0530 (7)	0.0703 (8)	0.0584 (7)	-0.0021 (6)	0.0148 (6)	-0.0092 (6)
C14	0.0606 (9)	0.0851 (11)	0.0969 (12)	-0.0146 (8)	-0.0120 (8)	-0.0230 (9)
C7'	0.0422 (6)	0.0431 (6)	0.0407 (6)	-0.0017 (4)	-0.0011 (4)	-0.0020 (4)
C8'	0.056 (6)	0.049 (7)	0.018 (5)	-0.011 (4)	-0.005 (4)	0.005 (4)
C9'	0.033 (3)	0.043 (4)	0.033 (5)	0.005 (2)	0.001 (3)	0.000 (4)
C10'	0.046 (3)	0.052 (4)	0.031 (4)	0.000 (2)	-0.003 (3)	0.007 (3)
C11'	0.039 (4)	0.047 (3)	0.027 (4)	-0.003 (3)	-0.004 (3)	0.008 (3)
C12'	0.047 (4)	0.053 (5)	0.024 (3)	0.010 (3)	-0.004 (3)	0.017 (3)
C13'	0.054 (5)	0.046 (5)	0.055 (6)	0.007 (3)	-0.022 (4)	0.015 (3)
O3'	0.042 (2)	0.070 (3)	0.032 (2)	0.009 (2)	0.0069 (17)	-0.0009 (18)
C15'	0.049 (3)	0.068 (4)	0.031 (3)	-0.015 (3)	0.002 (2)	0.001 (3)
C16'	0.0530 (7)	0.0703 (8)	0.0584 (7)	-0.0021 (6)	0.0148 (6)	-0.0092 (6)

Geometric parameters (Å, °)

O1—C7	1.2175 (13)	C13—H13	0.9500
O2—C2	1.3595 (16)	O3—C15	1.423 (3)
O2—C14	1.4300 (16)	C15—C16	1.516 (3)
C1—C2	1.3871 (16)	C15—H15A	0.9900
C1—C6	1.4293 (13)	C15—H15B	0.9900
C1—C7	1.5141 (15)	C16—H16A	0.9800
C2—C3	1.4130 (17)	C16—H16B	0.9800
C3—C4	1.353 (2)	C16—H16C	0.9800
C3—H3	0.9500	C14—H14A	0.9800
C4—C5	1.4054 (15)	C14—H14B	0.9800
C4—H4	0.9500	C14—H14C	0.9800
C5—C4 ⁱ	1.4054 (15)	C8'—C9'	1.384 (12)
C5—C6	1.438 (2)	C8'—C13'	1.402 (12)
C6—C1 ⁱ	1.4293 (13)	C9'—C10'	1.372 (12)
C7—C8	1.492 (3)	C9'—H9'	0.9500
C8—C9	1.377 (4)	C10'—C11'	1.395 (9)
C8—C13	1.391 (4)	C10'—H10'	0.9500
C9—C10	1.387 (4)	C11'—O3'	1.357 (8)

C9—H9	0.9500	C11'—C12'	1.371 (8)
C10—C11	1.390 (3)	C12'—C13'	1.401 (12)
C10—H10	0.9500	C12'—H12'	0.9500
C11—O3	1.365 (3)	C13'—H13'	0.9500
C11—C12	1.389 (3)	O3'—C15'	1.443 (7)
C12—C13	1.374 (4)	C15'—H15C	0.9900
C12—H12	0.9500	C15'—H15D	0.9900
C2—O2—C14	117.70 (12)	C12—C13—C8	120.7 (3)
C2—C1—C6	120.28 (11)	C12—C13—H13	119.7
C2—C1—C7	116.22 (10)	C8—C13—H13	119.7
C6—C1—C7	123.27 (10)	C11—O3—C15	117.9 (3)
O2—C2—C1	116.05 (11)	O3—C15—C16	106.77 (16)
O2—C2—C3	122.35 (11)	O3—C15—H15A	110.4
C1—C2—C3	121.52 (12)	C16—C15—H15A	110.4
C4—C3—C2	118.67 (12)	O3—C15—H15B	110.4
C4—C3—H3	120.7	C16—C15—H15B	110.4
C2—C3—H3	120.7	H15A—C15—H15B	108.6
C3—C4—C5	122.59 (11)	C15—C16—H16A	109.5
C3—C4—H4	118.7	C15—C16—H16B	109.5
C5—C4—H4	118.7	H16A—C16—H16B	109.5
C4—C5—C4 ⁱ	120.77 (15)	C15—C16—H16C	109.5
C4—C5—C6	119.61 (8)	H16A—C16—H16C	109.5
C4 ⁱ —C5—C6	119.61 (8)	H16B—C16—H16C	109.5
C1—C6—C1 ⁱ	125.46 (13)	C9'—C8'—C13'	114.9 (10)
C1—C6—C5	117.27 (7)	C10'—C9'—C8'	124.2 (9)
C1 ⁱ —C6—C5	117.27 (7)	C10'—C9'—H9'	117.9
O1—C7—C8	119.0 (2)	C8'—C9'—H9'	117.9
O1—C7—C1	119.18 (10)	C9'—C10'—C11'	118.1 (6)
C8—C7—C1	121.8 (2)	C9'—C10'—H10'	121.0
C9—C8—C13	118.9 (3)	C11'—C10'—H10'	121.0
C9—C8—C7	120.4 (3)	O3'—C11'—C12'	124.0 (9)
C13—C8—C7	120.7 (3)	O3'—C11'—C10'	114.1 (7)
C8—C9—C10	121.5 (3)	C12'—C11'—C10'	121.9 (6)
C8—C9—H9	119.3	C11'—C12'—C13'	117.1 (6)
C10—C9—H9	119.3	C11'—C12'—H12'	121.5
C9—C10—C11	118.8 (2)	C13'—C12'—H12'	121.5
C9—C10—H10	120.6	C12'—C13'—C8'	123.9 (8)
C11—C10—H10	120.6	C12'—C13'—H13'	118.1
O3—C11—C12	115.5 (3)	C8'—C13'—H13'	118.1
O3—C11—C10	124.3 (3)	C11'—O3'—C15'	119.3 (7)
C12—C11—C10	120.21 (18)	O3'—C15'—H15C	110.5
C13—C12—C11	119.9 (2)	O3'—C15'—H15D	110.5
C13—C12—H12	120.1	H15C—C15'—H15D	108.7
C11—C12—H12	120.1		
C14—O2—C2—C1	170.15 (11)	O1—C7—C8—C13	-10.0 (8)
C14—O2—C2—C3	-12.94 (18)	C1—C7—C8—C13	168.8 (5)

C6—C1—C2—O2	176.11 (8)	C13—C8—C9—C10	2.3 (10)
C7—C1—C2—O2	1.54 (14)	C7—C8—C9—C10	-176.7 (4)
C6—C1—C2—C3	-0.83 (16)	C8—C9—C10—C11	-0.7 (7)
C7—C1—C2—C3	-175.40 (11)	C9—C10—C11—O3	177.9 (3)
O2—C2—C3—C4	-174.32 (11)	C9—C10—C11—C12	-1.8 (4)
C1—C2—C3—C4	2.42 (18)	O3—C11—C12—C13	-177.0 (2)
C2—C3—C4—C5	-1.88 (17)	C10—C11—C12—C13	2.7 (3)
C3—C4—C5—C4 ⁱ	179.78 (13)	C11—C12—C13—C8	-1.2 (6)
C3—C4—C5—C6	-0.22 (13)	C9—C8—C13—C12	-1.3 (10)
C2—C1—C6—C1 ⁱ	178.75 (11)	C7—C8—C13—C12	177.6 (4)
C7—C1—C6—C1 ⁱ	-7.09 (7)	C12—C11—O3—C15	176.67 (16)
C2—C1—C6—C5	-1.25 (11)	C10—C11—O3—C15	-3.0 (3)
C7—C1—C6—C5	172.91 (7)	C11—O3—C15—C16	-173.45 (13)
C4—C5—C6—C1	1.79 (7)	C13'—C8'—C9'—C10'	-1 (4)
C4 ⁱ —C5—C6—C1	-178.21 (7)	C8'—C9'—C10'—C11'	0 (2)
C4—C5—C6—C1 ⁱ	-178.21 (7)	C9'—C10'—C11'—O3'	178.0 (8)
C4 ⁱ —C5—C6—C1 ⁱ	1.79 (7)	C9'—C10'—C11'—C12'	-1.2 (12)
C2—C1—C7—O1	109.85 (12)	O3'—C11'—C12'—C13'	-177.1 (7)
C6—C1—C7—O1	-64.54 (14)	C10'—C11'—C12'—C13'	2.0 (11)
C2—C1—C7—C8	-68.9 (4)	C11'—C12'—C13'—C8'	-2 (2)
C6—C1—C7—C8	116.7 (4)	C9'—C8'—C13'—C12'	2 (3)
O1—C7—C8—C9	168.9 (5)	C12'—C11'—O3'—C15'	8.7 (8)
C1—C7—C8—C9	-12.3 (9)	C10'—C11'—O3'—C15'	-170.5 (5)

Symmetry code: (i) $-x+5/4, -y+1/4, z$.

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg2 are the centroids of the C8–C13 and C1–C6 rings, respectively

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C16—H16C \cdots O1 ⁱⁱ	0.98	2.51	3.4919 (16)	175
C14—H14C \cdots Cg1 ⁱⁱ	0.98	2.83	3.716 (2)	151
C15—H15B \cdots Cg2 ⁱⁱ	0.99	2.80	3.6831 (19)	149

Symmetry code: (ii) $-x+1, y+1/4, z+1/4$.